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Energy Technology Division

Research Summary • 2001

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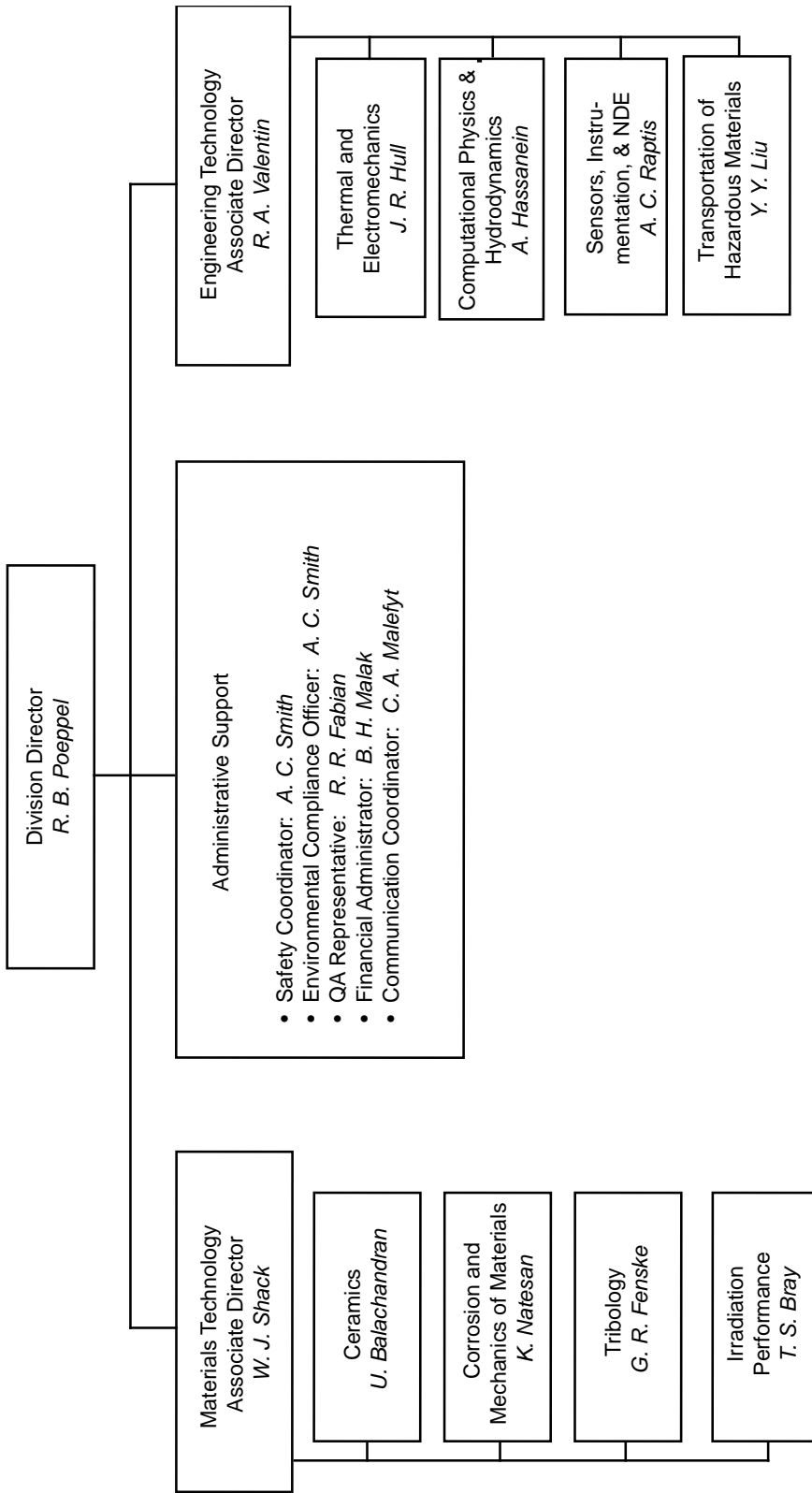
V. A. Martinez

April 2001

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Energy Technology Division

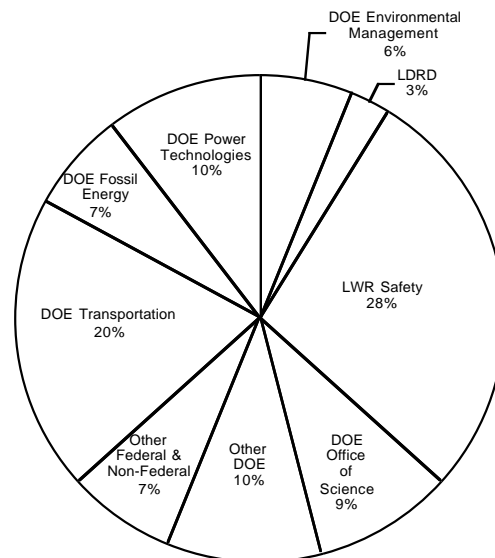


Overview of the Energy Technology Division

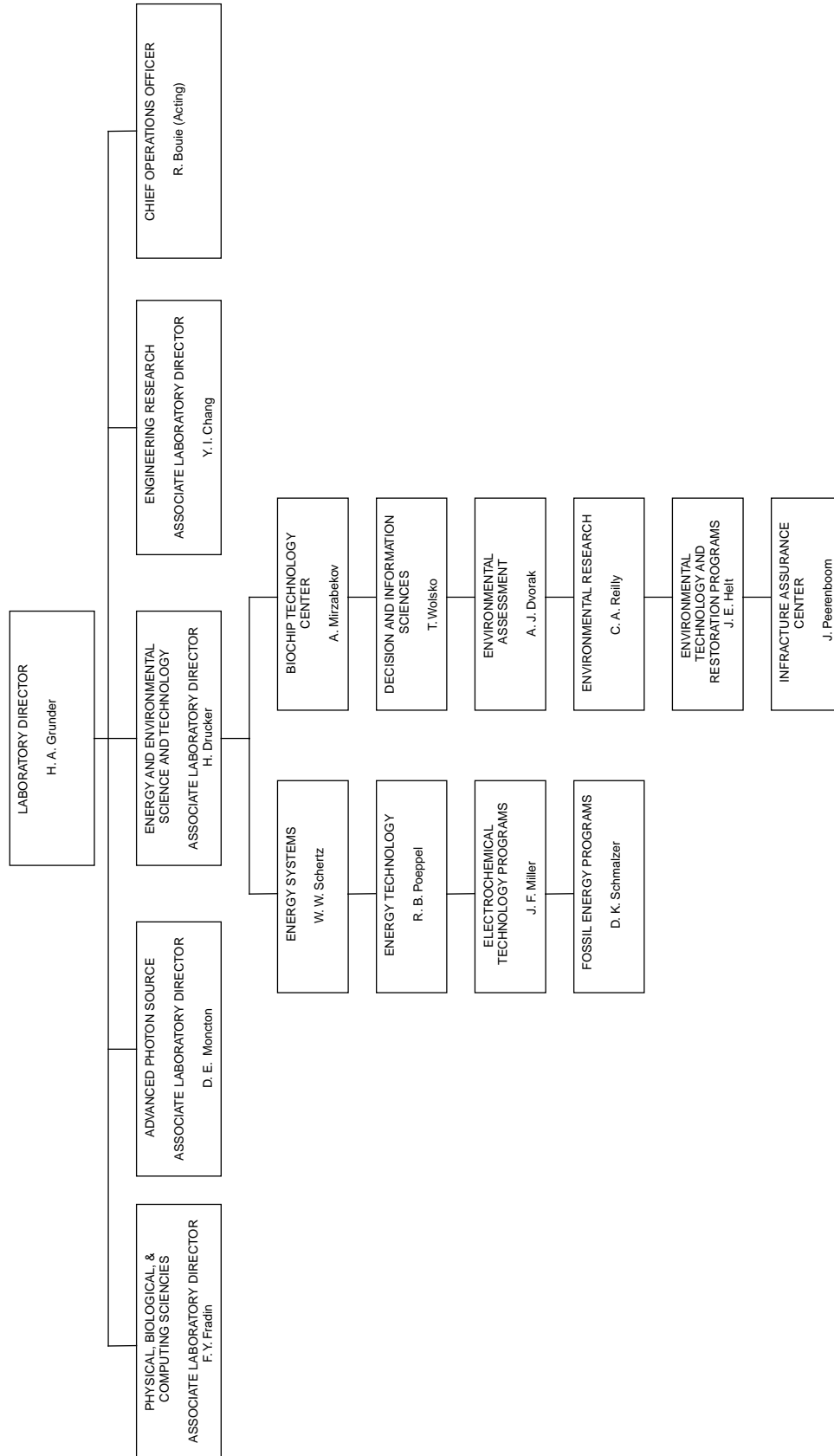
The Energy Technology Division provides materials and engineering technology support to a wide range of programs important to the U.S. Department of Energy. As shown on the preceding page, the Division is organized into eight sections, four with concentrations in the materials area and four in engineering technology. Materials expertise includes fabrication, mechanical properties, corrosion, friction and lubrication, and irradiation effects. Our major engineering strengths are in heat and mass flow, sensors and instrumentation, nondestructive testing, transportation, and electromechanics and superconductivity applications. The Division Safety Coordinator, Environmental Compliance Officer, Quality Assurance Representative, Financial Administrator, and Communication Coordinator report directly to the Division Director. The Division Director is personally responsible for cultural diversity and is a member of the Laboratory-wide Cultural Diversity Advisory Committee.

The Division's capabilities are generally applied to issues associated with energy production, transportation, utilization, or conservation, or with environmental issues linked to energy. As shown in the organization chart on the next page, the Division reports administratively to the Associate Laboratory Director (ALD) for Energy and Environmental Science and Technology (EEST). While most of our programs are under the purview of the EEST ALD, we also have had programs funded under other ALDs. Some of our research in superconductivity is funded through the Physical Research Program ALD. We also continue to work on a number of nuclear-energy-related programs under the ALD for Engineering Research. Under LDRD funding in several ET Sections, we are developing bioengineering programs and are attempting to obtain funding from NIH and DOE. Detailed descriptions of our programs on a section-by-section basis are provided in the remainder of this book.

This Overview highlights some major ET research areas. Research related to the operational safety of commercial light water nuclear reactors (LWRs) for the U.S. Nuclear Regulatory Commission (NRC) remains a significant area of interest for the Division. We currently have programs on environmentally assisted cracking, steam generator integrity, and the integrity of high-burnup fuel during loss-of-coolant accidents. The bulk of the NRC research work is carried out by three ET sections: Corrosion and Mechanics of Materials; Irradiation Performance; and Sensors, Instrumentation, and Nondestructive Evaluation.



ET Funding Sources for FY 2001



The Transportation of Hazardous Materials Section is the other main contributor to NRC programs; staff from that Section are preparing a Generic Aging Lessons Learned (GALL) report and the Standard Review Plan for License Renewal (SRP-LR) of operating nuclear power plants in the United States. The SRP-LR and the GALL report will provide guidance to NRC staff reviewers in performing safety reviews of license renewal applications in accordance with the license renewal rule. In addition to guidance development for plant license renewal, staff from the Section are also involved in the review of license renewal application for Calvert Cliffs, the first U.S. plant that received license extension for 20 years (in April 2000), and for Arkansas Unit-1, whose renewal application was submitted to the NRC in February 2000.

Superconductor development remains the largest individual program in the Division. The goal of this program is to develop ceramic superconductors for applications in generation, storage, transmission, and distribution of electrical energy. The general target is to produce in collaboration with industrial partners long lengths (>100 m) of conductor capable of carrying $>5 \times 10^5$ A/cm² at temperatures approaching that of liquid nitrogen. This work is being done in collaboration with American Superconductor (AMSC) and Intermagnetics General Corporation (IGC).

The Ceramics Section is developing dense ceramic membranes for separating hydrogen from gas mixtures. Because of concerns over global climate change due to carbon dioxide emissions, hydrogen is considered the fuel of choice for both the electric power and transportation industries.

Hybrid vehicles require a reduction in the size and weight of power electronic modules. To meet these needs, the Ceramics Section is developing high-dielectric-constant thin-film ceramic capacitors. In a collaborative effort among steel and refractory producers, universities, and national laboratories, the Ceramics Section is also developing refractory materials with improved performance and service life for use in electric arc furnaces.

The novel room-temperature-setting chemically bonded ceramic known as "Ceramicrete" is under development in the Division for stabilization of a wide range of DOE waste streams (such as contaminated ashes, salt cakes, and sludges). Ceramicrete also holds promise for treating waste streams generated by the utility, chemical, and defense industries. Several mixed wastes from DOE facilities such as the Savannah River Site, Fernald, Rocky Flats, Argonne East and West, and Idaho National Engineering and Environmental Laboratory have been successfully stabilized with Ceramicrete. The process has been scaled up to stabilize actual wastes; collaborations with private industry are ongoing to demonstrate the stabilization of DOE waste streams. Applications of Ceramicrete are also under development for oil-well borehole sealants.

The Corrosion and Mechanics of Materials Section addresses issues related to corrosion and the effects of various environments on mechanical behavior of materials used in several types of energy systems. Some of the key programs include development of electrically insulating coatings that are compatible in a lithium environment for fusion applications, mechanistic understanding of corrosion of metallic and ceramic materials in complex gas environments and in the presence of deposits for fossil applications, crack growth rate measurements and low-cycle fatigue properties of nuclear reactor materials for water reactor application, development of ultra-high-temperature intermetallics (supported by the

Synthesis and Processing Center of DOE/BES and Fossil Energy), and understanding the corrosion performance of waste containment materials in support of the Yucca Mountain project. During the past year, a new program (supported by the Office of Industrial Technologies of the DOE's Energy Efficiency and Renewable Energy branch) has been developed to gain a mechanistic understanding of the metal dusting phenomenon, a catastrophic degradation process that is widespread in several chemical and petrochemical processes.

Within the Tribology Section, development is continuing on near-frictionless carbon (NFC) coatings and assessment of their friction and wear properties for engine components (especially fuel injection systems) used in advanced internal combustion engines and fuel cell compressor/expanders intended for the Partnership for a New Generation of Vehicles (PNGV) program and for heavy vehicles. Basic research activities using Raman and NMR techniques are shedding light on the unique chemical bonding produced in the NFC coatings. Collaborative efforts with other federal laboratories and international laboratories confirm the results that we have reported on extremely low friction coefficients. Interest remains high, with more than 3,500 inquiries in the last three years. Approximately 15 Work-for-Others projects have been established to supply NFC-coated components for evaluation, and another 20 to 30 are being negotiated. The NFC coatings can now be produced by plasma-assisted chemical vapor deposition, a process that can be scaled up in size much more readily to handle parts in large quantities than can other approaches. CRADAs have been established to transfer this technology to large-scale coating systems. A commercial coating system is being procured; it will be modified for the NFC process and then used to address critical issues regarding coating uniformity in large-scale production scenarios.

The Japan Nuclear Cycle Development Institute is sponsoring work on the study of the mechanical properties of highly irradiated stainless steel structural materials from EBR-II. As part of this program, we have now developed the capability to remotely machine the test specimens and conduct mechanical testing in our hot cells. Such capabilities are available at probably only one other hot cell facility in the United States.

The Reduced-Enrichment Research and Test Reactor (RERTR) program seeks to develop Low-Enrichment Uranium (LEU, i.e., <20% ^{235}U enrichment) fuels to replace the High-Enrichment Uranium (HEU) fuels commonly used in research and test reactors around the world. A high-density U_3Si_2 dispersion fuel has been developed by ANL and approved by NRC and can be used to convert nearly 90% of the research reactors that use HEU. For some research and test reactors, however, the current loading density is not sufficient. To avoid derating these reactors, an advanced LEU fuel is necessary. Candidate fuel plates for irradiation testing are being designed and fabricated in the Division and will be examined and evaluated by us after irradiation.

Steam generator tubes account for more than 50% of the primary pressure-boundary surface of pressurized water nuclear reactors and have experienced in-service corrosive and mechanical degradation of various forms since the beginning of PWR commercial operations. ET is presently conducting the Steam Generator Tube Integrity Program under the sponsorship of the NRC to evaluate the integrity of nuclear steam generator tubes as plants age and degradation proceeds. An important activity under this program has been the design and construction of a tube mock-up that incorporates steam generator tubes containing prototypical flaws. This mock-up is being used to evaluate the reliability of current

nondestructive examination techniques for the detection and characterization of flaws. Major facilities have also been designed and constructed to determine failure pressures and leak rates for tubes containing a variety of flaws. Experimental data from these facilities are being compared to the predictions of analytical models also being developed as a part of this program. This research is being performed in ET's Corrosion and Mechanics of Materials; Sensors, Instrumentation, and Nondestructive Evaluation; and Thermal and Electromechanics Sections.

The Division continues to be actively involved in research to support the fusion reactor. Austenitic stainless steels, ferritic steels, and vanadium-base alloys are likely candidates for the first wall and divertor structural materials in these reactors. We are developing structural design criteria for these components; analyzing the damage that occurs in these components due to plasma instabilities; and determining the tensile, creep, fracture-toughness, and swelling properties of vanadium-base alloys after irradiation. Certain critical structural components in a fusion reactor must be cooled; advanced designs propose the use of Li or Pb-Li for this cooling. To mitigate liquid-metal magnetohydrodynamic (MHD) pressure effects in such systems, electrically insulating coatings on vanadium alloy structures are required, and studies are underway to develop a fundamental understanding of the critical materials science and Li-compatibility issues associated with such coatings.

The Computational Physics and Hydrodynamics Section is involved in several research areas such as magnetic fusion energy; inertial fusion, nuclear energy, and high energy physics programs; computational physics for medical applications, and other research projects. Most of these activities are based on the development of the comprehensive multidimensional HEIGHTS computer simulation package. The HEIGHTS (High Energy Interaction with General Heterogeneous Target Systems) package is a multipurpose computer package used to study various effects of particle beams and different radiation interaction with matter.

In magnetic fusion, for example, our research is oriented toward modeling the effects of plasma-material interactions during normal and abnormal operations. One important problem is modeling the effect of loss of plasma confinement on reactor first wall and divertor materials. For example, the intense energy deposited during plasma instability events over a short period can result in severe surface and bulk effects. Surface damage includes high erosion losses due to surface vaporization, spallation, and melt-layer erosion. Bulk damage effects include large temperature increases in structural materials and at the interface between surface coatings and structural materials. These large temperature increases will cause high thermal stresses, possible melting, and material fatigue and failure. Other bulk effects of some plasma instabilities, particularly those of longer duration or those with deeper deposited energy such as runaway electrons, can cause high heat flux levels at the coolant channels; this may cause burnout of these tubes and result in significant down-times for repair and maintenance. However, the initial stage of energy deposited during plasma instabilities is known to cause sudden formation of a vapor cloud above the exposed area. This vapor cloud, if well confined, may significantly reduce the net energy flux to the original disruption location, thus substantially reducing vaporization losses. Detailed physics of plasma/solid-liquid/vapor interactions in a strong and oblique magnetic field have been developed in the HEIGHTS package in a comprehensive self-consistent manner. Such detailed treatment of the magnetohydrodynamics and photon

radiation transport in the vapor-cloud region, for example, are found to be very important in determining the lifetime of plasma-facing components due to plasma instabilities.

The work of the Thermal and Electromechanics Section has been particularly successful in developing devices that utilize the unique properties of high-temperature superconductors in ways that have technological and industrial potential. Working closely with Commonwealth Research Corporation (CRC), the Section has developed and tested a series of flywheel energy storage devices that utilize very-low-loss superconducting bearings. The technology developed in this collaboration is now being transferred to Boeing in a DOE-funded program. This collaboration with CRC and Boeing is an excellent example of how the Division combines its technical expertise with that of private industry to move technology to the point of near-commercial deployment. Additional industrial collaboration within the Section relate to electromagnetic casting, development of fault-current limiters, improving the process for producing permanent magnets, and work on the cost-effective recycling of future automobiles.

The Division continues with its strong program on sensor development for a diverse group of DOE and industrial sponsors. The Sensors, Instrumentation, and Nondestructive Evaluation Section has unique strengths in millimeter-wave sensing, ultrasonics, and laser-based NDE systems. The Section is particularly good at developing a wide variety of industry-specific applications of a general technology. The work in acoustics/ultrasonic methods provides an excellent example. Basing its work on devices first used in fossil energy plants, the Section now has acoustic/ultrasonic technologies supported by the transportation industry for leak detection of components and engine control. Also, ultrasonic developments include temperature and pressure sensing, as well as sensing particulates in diesel engines. The ion mobility sensor work is focused on detecting NO_x in diesel auto exhausts. Millimeter-wave methods provide an excellent example of this. Basing its work on devices first used in the detection of chemical warfare agents, the Section now has millimeter-wave programs supported by the transportation industry for detecting and locating leaks in newly manufactured automotive components. Ultrasonics development ranges from indirect sensing of temperature in an advanced ceramic processing device to the sensing of particulates in diesel engine exhaust. In addition, the Section is developing programs in biosensing.

The Division has a long history of research and development of heat transfer and fluid flow systems for advanced energy systems. While much of this work grew out of nuclear reactor development programs, current emphasis has shifted to the chemical process and transportation industries. The programs of the Thermal and Electromechanics Section provide a good example of this transition. That Section has developed a strong program related to the behavior of compact heat exchange devices and has constructed an array of small general-purpose test facilities to study boiling, condensation, and flow visualization in near-commercial components of advanced energy systems. The current Section program includes design and testing of a unique dryer concept for the paper industry, the study of nanofluids and other novel coolants for automotive thermal management, and the application of phase-change systems such as pumped ice slurries in district cooling.

Although many of the Division's efforts are best described as advanced technology development projects that draw on a specific engineering or scientific discipline, we also have continuing programs that integrate a large number of specialized areas to provide a

unique group capability for a DOE sponsor. The Transportation of Hazardous Materials Section is the best example of this ongoing integrated approach to a specific DOE need. Section staff provide direct technical support to DOE in reviewing Safety Analysis Reports for Packaging (SARPs) of radioactive material shipping packages to ensure compliance with DOE, NRC, and DOT regulations. This work spans technical fields such as structural and thermal analysis, shielding and criticality, and operations, maintenance, and quality assurance. Working as a team on specific SARP submissions, staff provide DOE with independent technical review and confirmatory analysis needed to certify packages for shipment of a wide range of potentially hazardous nuclear materials. The work ensures worker safety, public health, and environmental protection and also affects DOE programs such as fissile material disposition, site closure, and waste disposal.

The Division continues its long history of research into advanced methods of simulation and modeling of fluid systems through the work of the Computational Physics and Hydrodynamics Section. While once almost completely supported by the Nuclear Regulatory Commission, the Section has shifted its priorities and now collaborates in projects with the auto industry and manufacturers of heavy equipment. A central focus has been to provide the auto industry with an advanced, general-purpose computational fluid mechanics code that can simulate the complex air flow and heat transfer associated with under-hood cooling and with passenger-compartment ventilation.

The Division's staff are active in national and international professional and scientific societies. They participate in joint programs, working groups, and national and international collaborations. They regularly attend meetings, present papers, organize conferences, and edit conference proceedings and journals. Some teach in local colleges and universities. Staff regularly receive awards and honors in recognition of their various achievements.

Engineering Technology

Computational Physics and Hydrodynamics

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The several research disciplines and modeling activities within the Computational Physics and Hydrodynamics Section include combined expertise in heat transfer, thermal hydraulics, atomic and plasma physics, magneto and shock hydrodynamics, radiation transport, and physics of material erosion and destruction, particle diffusion and mass transport, and computational fluid dynamics. One major activity is the study and simulation of materials behavior under intense power deposition for various national and international programs that are investigating magnetic fusion, inertial fusion, nuclear physics, high energy physics, space, medical, and industrial applications. In the reduced enrichment research test reactor (RERTR) fuel developmental program, the aim is to develop high-density, low-enriched uranium alloy fuels. A near-term goal of this modeling task is to provide an analytical interpretation of swelling in these materials as a function of irradiation condition and fuel composition. In computational fluid mechanics, the emphasis is on development and implementation of mathematical and phenomenological models of single-phase and two-phase fluids applied to a variety of advanced energy systems.

Modeling High-Power Interaction with Target Materials

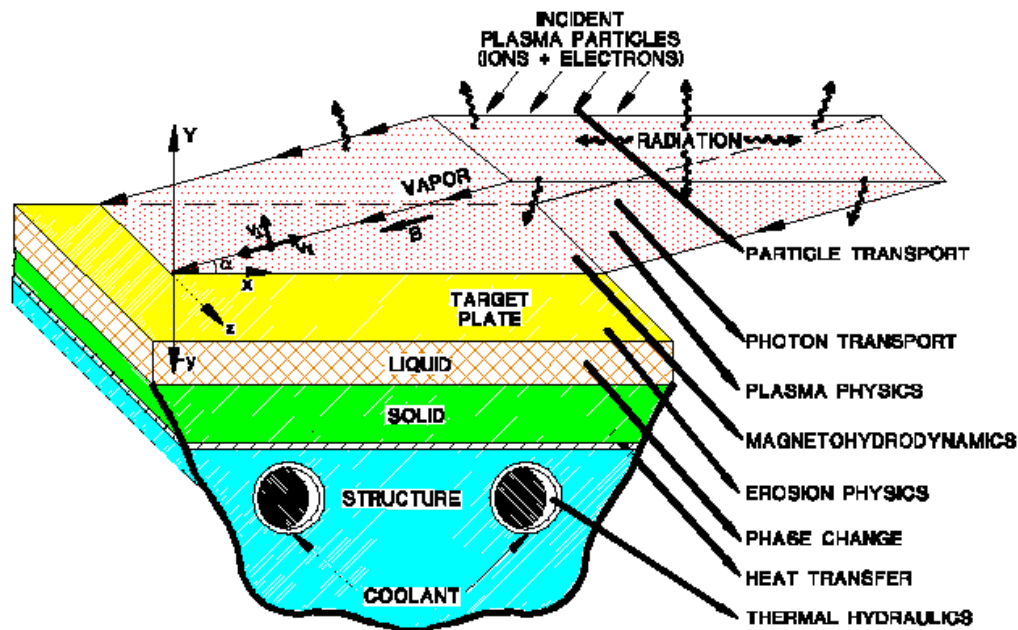
Much of this activity is based on development of the comprehensive multipurpose HEIGHTS (High Energy Interaction with General Heterogeneous Target Systems) package to study in detail the various effects of particle beams and radiation interaction with matter. This package is well known and has been used to model plasma-material interaction in powerful laboratory devices, as well as in tokamak machines in several countries such as Japan, Russia, and the United States.

In magnetic fusion, the Section's research activities are oriented toward modeling the effects of plasma/material interactions during normal and abnormal fusion reactor operations. One important problem is modeling the effect of loss of plasma confinement on reactor first-wall and divertor materials. For example, the intense energy deposited during plasma instability events ($10\text{-}200\text{ MJ/m}^2$) over a short period ($0.1\text{-}300\text{ ms}$) can result in severe surface and bulk effects. Surface damage includes high erosion losses due to surface vaporization, spallation, and melt-layer erosion. Bulk damage effects include large temperature increases in structural materials and at the interface between surface coatings and structural materials. These large temperature increases will cause high thermal stresses, possible melting, and material fatigue and failure. Other bulk effects of some plasma instabilities, particularly those of longer duration or those with deeper deposited energy such as that from runaway electrons, can cause high heat flux levels at the coolant channels; this may cause burnout of these tubes and result in significant down-times for

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²Serves two sections.

repair and maintenance. However, the initial stage of energy deposited during plasma instabilities is known to cause sudden formation of a vapor cloud above the exposed area. This vapor cloud, if well confined, may significantly reduce the net energy flux to the original disruption location, thus substantially reducing vaporization losses. Detailed physics of plasma/solid-liquid/vapor interactions in a strong and oblique magnetic field have been developed in the HEIGHTS package in a comprehensive self-consistent manner, as schematically illustrated in the figure below. Such detailed treatment of the magnetohydrodynamics and photon radiation transport in the vapor-cloud region, for example, is essential for determining the lifetime of plasma-facing components due to plasma instabilities.



Required integrated modeling areas for comprehensive evaluation of damage from loss of plasma confinement

In the inertial fusion program, we are modeling the effects of pellet/target implosion and resulting debris on the dynamic response of the reactor chamber walls between microexplosions.

In the nuclear physics program, our analysis is aiding in design of a viable target system and the study of beam-on-target interaction for the Rare Isotope Accelerator (RIA). We use the 3-dimensional (3-D) HEIGHTS package to calculate the thermal response of different target system cooled by liquid metals as the particle beam penetrates the composite target system.

In the high-energy physics program, we are modeling the target response of the international muon collider and the neutrino factory projects. This requires studying the penetration of free high-velocity (10-20 m/s) liquid-metal jets in a strong magnetic field of 20 T. We then study the shock hydrodynamic effects due to proton beam bombardment for the production of pions that decay to muons.

Another recent application of the HEIGHTS package is the study, for medical applications, of the effects of electric shock and injuries resulting from electrical arc explosions.

HEIGHTS can also be used to simulate and understand the processes through which granulated metal nanostructures can be fabricated based on laser ablation under the influence of a pulsed laser beam. Liquid metal droplets of submicrometer size are first splashed out from the melted target surface and then, following charging in a laser-induced plasma, break up to form a nearly monodispersed flux of charged nanometer-sized droplets. This flux is forced to the substrate by an electric field, and the deposited structure is self-assembled in a densely packed amorphous layer due to Coulomb interaction. This work is in collaboration with experimentalists at Ioffe Institute in St. Petersburg, Russia.

In summary, HEIGHTS is a comprehensive software package that can run on computers ranging from parallel processors to Cray supercomputers to engineering workstations. HEIGHTS combines the foremost numerical solution methods including finite-element, Lagrangian, Eulerian, Particle-in-Cell (PIC), Monte Carlo, and Ray Tracing Techniques. Depending on the problem's complexity, run-time ranges from one hour to several months.

Modeling Irradiation Behavior of Uranium-Alloy Fuels for the RERTR

The fuel developmental program is aimed at development of high-density, low-enriched uranium alloy fuels for RERTR. An experimental fuel matrix has been developed and the results from the first three irradiations in the Advanced Test Reactor (ATR) are currently being assessed. The modeling subtask is fully integrated with the experimental program. A near-term goal of the modeling task is to provide an analytical interpretation of swelling in these materials as a function of irradiation condition and fuel composition. The modeling task is aimed at developing mechanistic models describing specific phenomena (e.g., irradiation-induced recrystallization), as well as the development of a fuel performance code (DART) for full-scale fuel plate (or tube or rod) in-reactor simulations.

A rate-theory-based model is developed to investigate the nucleation and growth of interstitial loops and cavities during low-temperature in-reactor irradiation of uranium-molybdenum alloys. Consolidation of the dislocation structure accounts for generation of forest dislocations and capture of interstitial dislocation loops. The theoretical description includes stress-induced glide of dislocation loops and accumulation of dislocations on cell walls. The loops accumulate and ultimately evolve into a low-energy cellular dislocation structure. Calculations indicate that nanometer-size bubbles are associated with the walls of the cellular dislocation structure. The accumulation of interstitial loops within the cells and of dislocations on the cell walls leads to increasing values for the rotation (misfit) of the cell wall into a subgrain boundary and a change in the lattice parameter as a function of dose. Subsequently, increasing values for the stored energy in the material are shown to be sufficient for the material to undergo recrystallization. Results of the calculations (e.g., for change in lattice parameter, dislocation density, bubble-size distribution) have been successfully compared with SEM photomicrographs of irradiated U-10Mo, as well as with data from irradiated UO_2 .

A theoretical model is also developed for the interdiffusion of Al and U alloys. Data on the interdiffusion of Al and uranium silicide during irradiation have resulted in an empirical

correlation that expresses the thickness of the interaction zone as linearly dependent on the fission rate with exponential temperature dependence. Recent data for the interdiffusion of Al and U-10Mo is consistent with this correlation. However, these dependencies have no physical basis. In general, at the low temperatures of interest for RERTR, radiation-enhanced diffusion is dominant over thermal diffusion. In turn, the specific mechanism of radiation-enhanced diffusion (e.g., of Al in UAlx) is dependent on the defect properties in the material. If the defect lifetimes are dominated by pair annihilation, then radiation-enhanced diffusion is proportional to the square root of the fission rate with exponential temperature dependence. On the other hand, if the defect lifetimes are dominated by their interaction with sinks (e.g., a fixed density of dislocations), then the predicted dependence is linear with fission rate and is athermal.

A model for the interdiffusion of Al and U alloys has been developed on the basis of the premise that generation of a high dislocation density leading to the formation of a cellular dislocation network provides a sufficient sink density in the material such that the sinks dominate defect behavior. Most models for irradiation-enhanced diffusion appearing in the open literature treat the sink density as constant and independent of temperature. However, in general, the steady-state dislocation density is dependent on temperature.

As part of a Sisterlab agreement, ANL and Comisión Nacional de Energía Atómica (CNEA) continued to collaborate on the generation of a user-friendly PC version of the DART code that incorporates a visual interface to expedite the extraction of calculated data. The generation of this new version of DART will include modification of existing models, implementation of new models, and construction of a new graphical I/O interface such that the user/code dialog becomes highly optimized. The eventual conversion of DART to a parallel architecture is expected to facilitate its potential development. In addition, work continues within the ongoing collaboration between Bochvar Institute, Russia, and ANL on the analysis of new high-density uranium alloy fuels for the RERTR. Substantial progress has been made in assessing the performance of U-Mo alloy fuel.

Computational Fluid Mechanics

Emphasis in this work is on development and implementation of mathematical and phenomenological models of single-phase and two-phase fluids applied to a variety of advanced energy systems. In the past, the Section has developed models related to components and safety systems of advanced nuclear reactors. In recent years, however, work in this area has ended and several new classes of simulation have gained importance. In a cooperative program with private industry, computer methods to model the fluid mechanics of casting and welding processes received considerable emphasis and led to the development of the Casting Process Simulation (CaPS) software package. Current work within the Section has shifted into code development for automotive applications, for two-phase flow simulation studies, and for specialized process modeling applications.

Application of High-Performance Computing to Automotive Design and Manufacture

The Section has participated with members of several other ANL divisions in a collaborative project with the Big Three auto makers to develop a general-purpose computational fluid mechanics (CFD) code applicable to problems in combustion, underhood

cooling and HVAC, and exterior aerodynamics. This activity has been built on a code developed at Los Alamos National Laboratory, that is, the CHAD (Computational Hydrodynamics for Advanced Design) code. During the past year, ANL's focus has been on underhood cooling and on developing CHAD modifications that would model behavior of the radiator, fan, and other components.

Several components in automotive systems have characteristic structures smaller than can be practically resolved through conventional modeling. Examples include filters, vanes, heat exchangers (radiator, heater core, evaporator, etc.), mufflers, catalytic converters, and screens. These vehicle components are present in fuel, lubrication, cooling, and exhaust systems. Consider a CFD model for a catalytic converter: while theoretically possible to model the full flow field, it is not practical to assume that the detailed flow, temperature, and concentration fields of the exhaust gases flowing around the multitude of spheres comprising the catalytic medium can be modeled by accounting for each of the spheres with complete boundary conditions. To handle such situations, a porous-medium formulation is being considered.

The porous-medium formulation can include the parameters of volume porosity, directional surface permeability, distributed mass source or sink, distributed resistance, and distributed heat source or sink. With this formulation, an anisotropic flow domain with stationary structures can be modeled. The porous medium formulation with directional surface permeability represents a unified approach to thermal-hydraulic analysis. Surface permeability is the ratio of fluid flow area through a control surface to the total control surface area. Volume porosity is similarly defined as the ratio of the volume occupied by fluid in a control volume to the total control volume. Accurately accounting for these geometrical aspects reduces the required accuracy for the correlations needed in the distributed mass, momentum, and energy source terms. The distributed mass source or sink accounts for the effect on species concentration due to the fluid interacting with the immersed solid structures. The distributed resistance is a drag force opposing the flow and is due to the fluid flowing around the immersed solid structures. The distributed heat source or sink represents the thermal interaction with the immersed solid structures.

It has been determined that a complete implementation of the porous medium formulation is beyond the scope of the current automotive project. Much of CHAD, as well as preprocessors, would have to be modified or rewritten to account for the geometrical aspects. Therefore, a partial implementation consisting of only the concept of distributed resistance has been implemented and tested.

Automotive Underhood Thermal Management Analysis with 3-D Coupled Thermal-Hydrodynamic Computer Models

Argonne National Laboratory and Lockheed Martin Energy Research Corporation, Analysis and Design Application Co., Ltd. (adapco), and the Supercomputer Automotive Applications Partnership (SCAAP) formed under the United States Council on Automotive Research (USCAR) have entered into a Cooperative Research and Development Agreement (CRADA). The purpose of this CRADA is to develop an Underhood Thermal-Hydrodynamic Integrated Model for the analysis of coupled thermal-hydrodynamic underhood phenomena. The model will be based on the CHAD code, which has been developed specifically for the analysis of hydrodynamic phenomena in complex geometries and has been selected as the

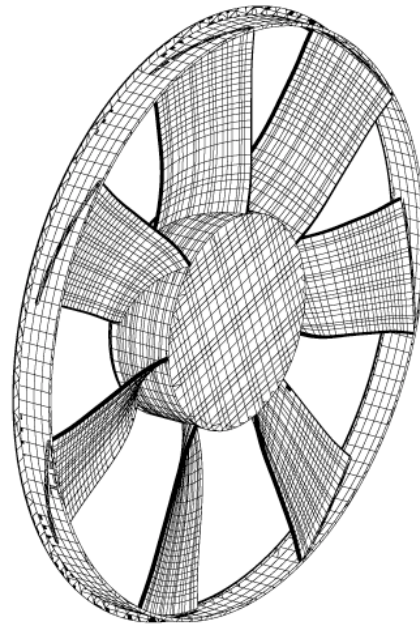
analysis tool by USCAR and SCAAP. The focus is to develop and integrate thermal models for convective, conductive, and radiative heat transport, and to develop models for critical heat management system components including fans and heat exchangers. Substantial effort is placed on improving the numerical solution algorithms for modeling the behavior of 3-D thermal-hydraulics systems on state-of-the-art massively parallel computers. Extensive verification and validation of the resulting models will be performed, through comparison to detailed thermal and flow measurements and to industry-standard computational solutions. The major ET focus is on testing a simplified fan model and developing a heat exchanger model for use in this project.

Fan Model Development and Testing

Simulation of a fan by direct solution of the Navier-Stokes equations in a moving grid requires a significant effort for grid generation and very long computational time. For design analyses that require underhood thermal management simulations, a simpler but adequately accurate fan model is needed. The objective of this work is to develop such a model that will be used to predict velocity distributions past the blades of a fan. These velocities will be used either as a velocity source or to generate body force sources for the computational cells occupied by the fan blades. These sources will then be used in CHAD and in adapco's STAR-CD code to simulate the effects of the fan in an underhood flow field.

In this simple model, the actual fan is approximated by another fan (actuator disk) having the same diameter and a very large number of very narrow equal blades such that at any radius both fans have the same solidity. Using the actuator disk and blade element theory of a propeller, a set of equations has been derived to provide the circumferential and axial velocity of the fluid past the blade of the fan. These equations require as input the rotational speed of the fan, geometric fan data, and lift and drag coefficients of the blades. To determine these coefficients, the blade is cut into a number of sections (the points of each section have the same radius). Then, each section is approximated with an airfoil whose drag and lift coefficients are known. This model has been successfully coupled with the CFD code STAR-CD.

To validate the ANL fan model, this model coupled with STAR-CD was used to analyze a fan experiment performed by Daimler Chrysler (see next figure). In this experiment, the fan has seven identical blades arranged symmetrically. Each blade is a cambered plate of constant thickness. With the aid of PROSTAR (preprocessor and postprocessor code for geometrical modeling and mesh generation), geometrical information such as the chord, the thickness, cambered distance and blade angle at any radius can be obtained. The radius at the root of the blade was 0.081 m



Model fan geometry used in CFD codes

and the radius at the tip of the blade was 0.221 m. In the present analysis, a total of 24 radii are used to obtain the geometrical information of the blades. Air velocities are measured downstream of the fan at various distances from the fan, which operated at 2400 rpm. The velocity measurements closest to the fan (25 mm) are most relevant for validation of the fan model.

Information from the ANL fan model is used in the CFD code STAR-CD to represent the fan as either a “velocity” source or a body-force source. In the first case, in the computational cells of the STAR model that are occupied by the fan, the air velocities were fixed to the values predicted by the simple model. In the second case, a body force was introduced with magnitude and direction determined by a simple model. The calculated velocity distribution of air in the vertical plane passing through the center of fan is calculated. For these predictions, the first-order upwind scheme was used in the approximation of the convective terms of the Navier-Stokes equations, and turbulence was simulated using the high-Reynolds-number κ - ϵ model. Agreement between predictions and measurements is good, especially in the representation of the fan as a body force.

Heat Exchanger Model Development

Development of the heat exchanger model is focused on the radiator, a liquid-to-air heat exchanger. Hot liquid flows from the engine and enters the radiator coolant passages. Air flowing through the outside of the radiator removes the heat.

The current state-of-the-art radiator model used by industry has been identified as the radiator model used in adapco’s STAR-CD code and by the automobile manufacturers. An equivalent radiator model for the CHAD code is being developed.

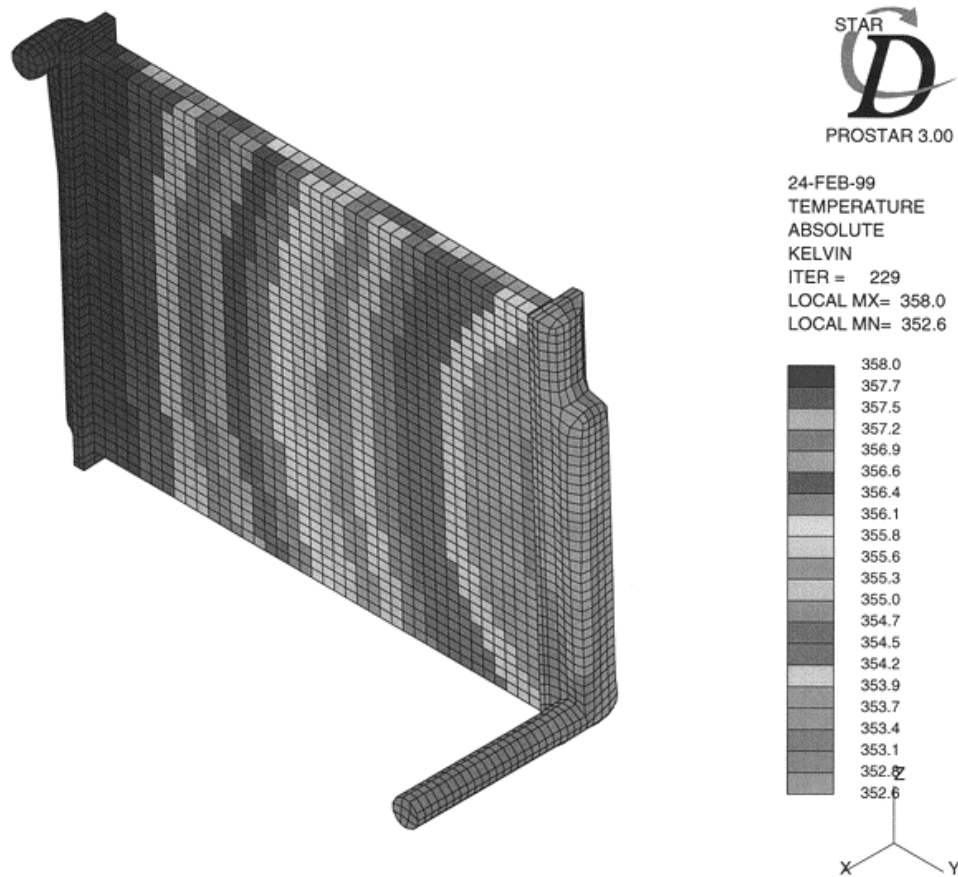
Development of a state-of-the-art heat exchanger model for CHAD is continuing. While the initial application is focused primarily on a radiator-type heat exchanger, the implementation is general enough to accommodate any number of heat exchangers in a simulation. The concept of mesh-matched core regions is used to thermally link the two fluid streams.

A simple heat exchanger with an air-side core region and a coolant-side core region is being used to test and debug the new code as the new capabilities are implemented into CHAD. Initial testing has been completed successfully. Next, a more realistic model of a radiator will be constructed and simulated.

The next figure shows the temperature distribution of the coolant in a radiator, using model data provided by adapco.

Applications of COMMIX in the Paper Industry

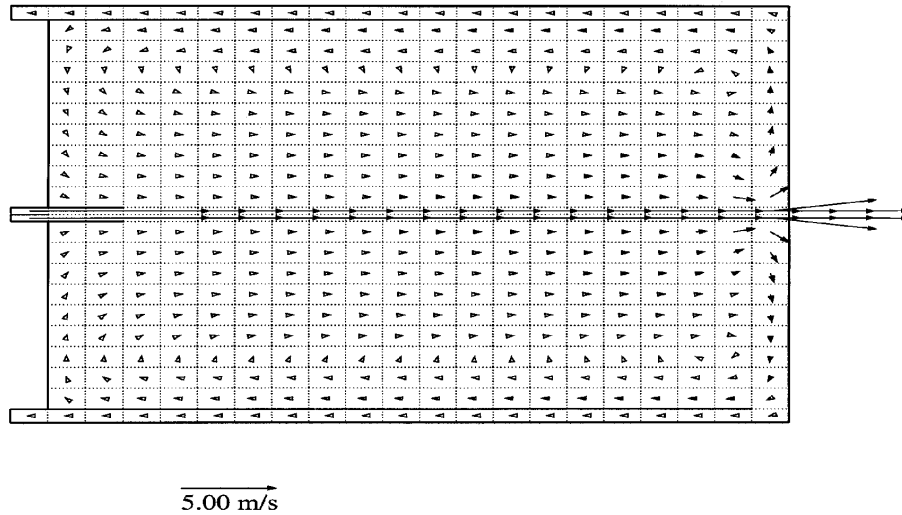
For many years, the Section has been concerned with models of complex flows within large vessels and between interconnected vessels of various geometries. Almost all of these simulations were related to nuclear reactor safety problems; however, in terms of the generic class of components for which the COMMIX code was designed, such simulations could



Temperature distribution of coolant inside a radiator

apply equally well to process simulation problems in the paper industry. In an attempt to show COMMIX as a good tool for the paper industry, the Section has performed several sample calculations related to steam condensation on the inside wall of a cylinder dryer.

The next figure, typical of these simulations, shows the flow distribution within a specific cylinder dryer. Here, the saturated steam enters horizontally through the center of the left surface of the cylinder dryer. When the steam flow reaches the opposite end of the cylinder, it deflects and flows radially outward along the cylinder surface. The flow then turns horizontally toward the left along the cylinder wall when it reaches the corner. Due to the presence of a channel partition, the leftward horizontal flow is separated into two streams. One stream flows along the inner wall of the partition and the other into the channel formed from the partition and the cylinder wall. The stream that flows along the inside partition turns radically inward when it reaches the left end of the cylinder, and then turns horizontally at the center to join the inlet stream to form a recirculating zone. The steam that enters the channel continues to flow and deposit heat to the wall to dry the paper on the outside and finally exit to the left. Part of the steam condenses and forms a liquid film on the cylinder wall, resulting in increased thermal resistance and decreased heat transfer. If the steam velocity inside the channel is sufficiently high, part of the condensate is then carried out by the remaining steam while it blows through the channel. As a result, the thickness of the liquid film is reduced and the heat transfer rate is increased.



Velocity field on symmetrical plane of a cylinder dryer

The issue of interest in this simulation was how certain channel geometry changes such as the distance between the cylinder wall and the channel partition would alter the steam condensation rate for a fixed inlet steam flow rate. The results of these COMMIX calculations were used initially by the Thermal and Electromechanics Section to design the channel geometry for an optimal stationary cylinder dryer.

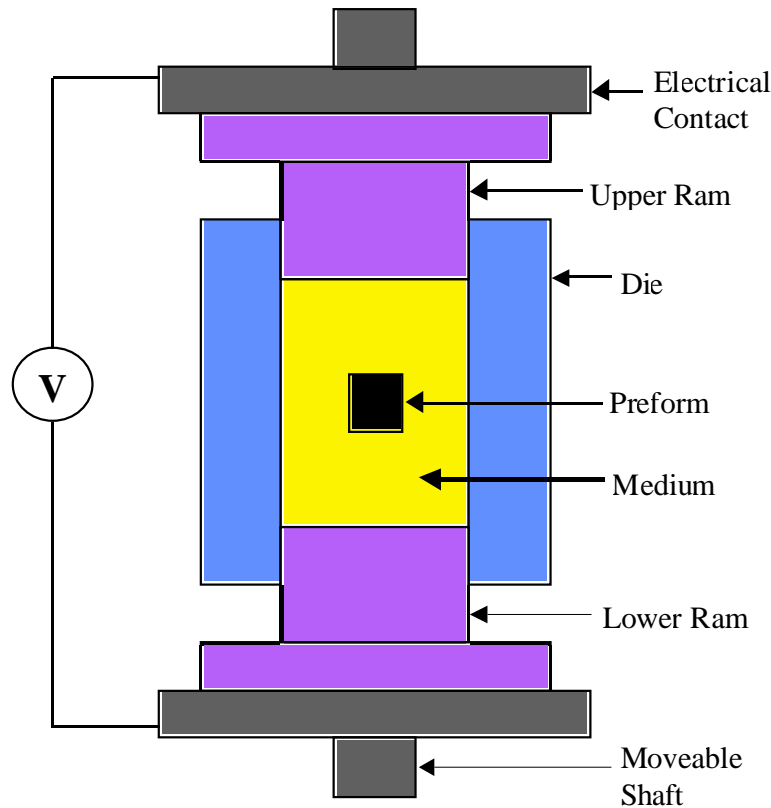
Modeling of Electroconsolidation

Electroconsolidation, a proprietary process developed by Superior Graphite Co., provides rapid, pressure-assisted densification of preformed materials. It can be used to manufacture parts from powders, including those of metals, ceramics and polymer materials. It offers advantages when pressure is necessary or desirable while heating the part to be consolidated. Preformed materials of complex shape can be densified directly to near-net-shape articles by the process.

The part to be densified is immersed in a bed of special free-flowing granular media within a cylindrical container or die chamber. Pressure is applied by rams acting on the bed (see next figure). Electric current heats the granular media, and heat is transferred from the media to the part by conduction while the part is subjected to compaction pressure.

Electroconsolidation is one of a class of “soft-tooling” processes that use particulate solids as “pseudo-fluid” for transmitting pressure to a workpiece. It differs from other processes because the granular pressure-transmitting media is heated while in direct contact with the workpiece. Moreover, the “inside-out” manner of heating the workpiece enables ultrarapid heating and the ability to achieve controlled temperatures above 2500°C. The result is a very short cycle time.

The purpose of this three-year project, funded by the National Institute of Standards and Technology’s Advanced Technology Program (ATP), was to develop a computer model of the pressure and temperature distributions inside the die to serve as the basis of the



Schematic diagram of die assembly used in Electroconsolidation

process control system. The model allows calculation of the workpiece temperature and can be used to help determine the best positions for multiple workpieces in the die to minimize temperature differences among them. Two existing computer codes were used for the Electroconsolidation process and were coupled to produce the desired result. ELEKTRA is a commercial code for 3-D electromagnetic-field calculation; no direct code modification is possible. The second code, MaPS, is a 3-D materials process simulator and is a derivative of CaPS directly modified for this application; the modifications are done in five stages so that each can be verified experimentally before proceeding to the next stage.

The computer model for temperature prediction contains three modules. The first (the density module) calculates the density and resistivity of the particulate medium as a function of pressure in the die. Resistivity at each position is fed into the second module (the resistivity-heat module), where the rate of resistive heating is calculated in ELEKTRA. That information, along with heat transfer calculations, is used by the third module (the temperature module) in MaPS to predict the temperature at any location in the system as a function of time.

Numerical simulations of resistive heat generation and temperature distribution within the die chamber have been performed in the Electroconsolidation process. Coupling of ELEKTRA and MaPS has provided encouraging consistency and a reasonably accurate prediction of the pressure, heat transfer, and temperature profiles within an Electroconsolidation die. The developed computer model provides a better understanding of the entire Electroconsolidation process. The model demonstrates the feasibility of a

proprietary process for achieving simultaneous application of pressure and heat to powder densification in Electroconsolidation.

In addition to being the basis for a real-time feedback control system, the computer model can be used to estimate power and time requirements in Electroconsolidation. It can also be used to plan the placement of multiple preforms in the die to minimize temperature differences during densification and therefore optimize the overall Electroconsolidation operation.

Modeling of MHD for Industrial Processes

We have unique capabilities that are ideally suited to the application of numerical MHD technology in industrial processes. The Laboratory's broad expertise in MHD has been acquired over many years; we have extensive experience in the numerical simulation in electric and magnetic fields, thermal hydraulics, casting process, and performance evaluation. Several developed technologies are described below.

Electromagnetic (EM) Stirring

We worked with the Thermal and Electromechanics Section and J. Mulcahy Enterprise, a manufacturer of EM stirring equipment, in the development of a numerical model to model a novel EM stirring system that uses two rotating magnetic fields. The system stirs a flow by a main EM stirrer (M-EMS), and control is achieved by applying to the meniscus region an auxiliary EM field whose direction of rotation is opposite to that of the M-EMS. Numerical simulations of EM fields and fluid flows induced by them within metal pools had been performed for cylindrical and square geometry that closely approximates an actual continuous casting arrangement. The shape of the free surface between liquid metal and air can be tracked by introducing a volume tracking method in the model.

Numerical simulations were performed to compute the electromagnetic force and fluid flow by coupling the finite-element electromagnetic code ELEKTRA and the finite-difference thermal hydraulic code CaPS. ELEKTRA solves 3-D time-varying electromagnetic field equations and predicts the induced eddy currents and electromagnetic forces. The time variation can be either transient or steady-state AC. CaPS provides an efficient solution of transient heat conduction within the metal and between the metal and the mold and computes the profile of the free surface. The computed 3-D magnetic fields and induced current densities in ELEKTRA are used as input to flow-field computations in CaPS. The model involves the solutions of the Maxwell equations, the Navier-Stokes equations, and the transport equations for the turbulence kinetic energy k and its rate of dissipation. Turbulent flow is included to describe recirculating electromagnetically induced flows, and control of turbulent flow in the pool is an efficient method to improve performance of the EMS system.

The model has computed values and spatial distributions of electromagnetic parameters and fluid flow in the stirred pools of mercury in cylindrical and square geometry. Also predicted were the relationships between electromagnetics and fluid flows pertinent to a dynamic equilibrium of the opposing stirring swirls in the meniscus region. The computer model provided encouraging consistency and reasonable accuracy in predicting swirl flows induced by the combination of a main stirrer and an auxiliary inductor. The developed model

demonstrated validity in predicting stirring flow control in a continuous casting mold based on EM parameter inputs.

Electromagnetic (EM) Containment

Traditionally, most thin steel sheets are made by continuous casting of 50- to 300-mm-thick slabs, followed by hot rolling to reduce the thickness to 2.5 mm and then cold rolling to the final thickness. The hot rolling stage is very capital- and energy-intensive, adding significantly to the cost of the finished product. To develop the capability for casting relatively thin sheets of steel in this collaborative effort, a device was designed to form a continuous thin steel sheet between two counter-rotating rollers. In such twin-roll casting, thin sheets can be cast by eliminating the hot-rolling process, and the resulting cost savings can give the sheet product an enormous economic advantage over products made by competing methods. However, ceramic dams in twin-roll casting have only a short lifetime because they are susceptible to erosion and breakage. They also are sites at which the molten metal can solidify and become attached to the strip being formed, thus altering the roll gap spacing and hence the thickness of the cast product and the surface temperature. These alterations can lead to surface defects, variation in product thickness, leakage of liquid steel from the caster, and even strand breakage and liquid steel breakouts. Therefore, the use of EM fields was proposed to produce EM force to contain liquid metal in the moldless twin-roll casting. The application of EM edge dams (EMDs) in twin-roll casting can bypass the fundamental problems of ceramic solid dams and thus make economic sense. Application of EM fields in twin-roll casting is a complicated process that involves interaction among electric, magnetic, thermal, mechanical, and metallurgical phenomena. Therefore, modeling is needed to help optimize the performance of products, enhance their value, get them to the marketplace sooner, and gain competitive advantages for business and industry.

We have worked together with ET's Thermal and Electromechanics Section and Inland Steel Company to develop a 3-D computer model that can predict fluid flows, eddy currents, liquid-metal containment, and shape of free surface in caster designs. Numerical simulations were performed to compute the EM force and fluid flow by coupling the finite-element EM code ELEKTRA and the finite-difference casting process code CaPS-EM. ELEKTRA solves 3-D time-varying EM field equations and predicts the induced eddy currents and EM forces. CaPS-EM provides an efficient solution of transient heat conduction within the metal and between the metal and the mold and computes the profile of the free surface.

The developed model has provided encouraging consistency and a reasonably accurate prediction of the flow pattern, free surface shape, and EMD containment. It demonstrates the feasibility of full-face containment by the EMD under various operating conditions. The developed mathematical model can greatly shorten casting research related to use of EM fields for liquid-metal containment, stirring, and control. The model also can optimize the existing casting processes and minimize expensive and time-consuming full-scale testing. The model has been used to study the complicated magnets for edge dams of twin-roll casters.

Recent Work by the Computational Physics and Hydrodynamics Section

Publications

A Coupled Newton-Krylov Solver for Improved CHAD Cache Utilization

T. R. Canfield, T. H. Chien, H. M. Domanus, A. M. Tentner, C. P. Tzanos, R. A. Valentin, and D. P. Weber

Presented at Advanced Simulation Technologies Conf. (ASTC 2000), High Performance Computing Symp., Washington, DC, April 16-20, 2000, ANL/MCS-P800-0300.

Computer Modeling of Temperature Prediction for Electroconsolidation

F. C. Chang; R. R. Fessler (Biztek Consulting, Inc.); B. D. Merkle, J. M. Borton, and W. M. Goldberger (Superior Graphite Company)

Paper presented at 2000 Fall Mtg. of TMS, St. Louis, Oct. 8-12, 2000.

Numerical Simulation of MHD for Electromagnetic Edge Dam in Continuous Casting

F. C. Chang

Proc. 15th Intl. Conf. on Advanced Science and Technology (ICAST '99), Argonne National Laboratory, April 2-3, 1999, pp. 171-182.

Modeling of MHD Edge Containment in Strip Casting with ELEKTRA and CaPS-EM Codes

F. C. Chang

Argonne National Laboratory Report ANL-00/1.

Simulation of Intense Heating and Shock Hydrodynamics in Free-Moving Liquid Targets

A. Hassanein

Paper presented at the Nuclear Applications of Accelerator Technology, ACCApp '99, ANS Mtg., Long Beach, CA, Nov. 14-18, 1999.

Irradiation-Induced Recrystallization of Cellular Dislocation Networks in Uranium Molybdenum Alloys

J. Rest and G. L. Hofman

Paper presented at Materials Research Society Fall Meeting, Boston, Nov. 27-Dec. 1, 2000.

FASTDART: A Fast, Accurate and Friendly Version of DART Code

J. Rest and H. Taboada (Comision Nacional de Energia Atomica)

Paper presented at 23rd Intl. Mtg. on Reduced Enrichment for Research and Test Reactors, Las Vegas, Oct. 1-6, 2000.

A Theoretical Model for the Irradiation-Enhanced Interdiffusion of Al and U Alloys

J. Rest

Paper presented at 23rd Intl. Mtg. on Reduced Enrichment for Research and Test Reactors, Las Vegas, Oct. 1-6, 2000.

Calculation of the Evolution of the Fuel Microstructure in UMo Alloys and Implications for Fuel Swelling

J. Rest, G. L. Hofman; I. Konovalov, and A. Maslov (Bochvar Inst.)
 Proc. 22nd Intl. Mtg. on Reduced Enrichment for Research and Test Reactors
 (RERTR), Budapest, Oct. 3-8, 1999.

Theory and Models of Material Erosion and Lifetime during Plasma Instabilities in a Tokamak Environment

A. Hassanein and I. Konkashbaev
 Fusion Engineering and Design 51-52 (2000) 681-694.

Hydrodynamic Effects of Eroded Materials of Plasma-Facing Component during a Tokamak Disruption

A. Hassanein and I. Konkashbaev
 J. Nucl. Mater. 283-287 (2000) 1171-1176.

Liquid Metal Targets for High-Power Applications: Pulsed Heating and Shock Hydrodynamics

A. Hassanein
 Laser and Particle Beams (2000), 18, 1-12.

Comprehensive Physical Models and Simulation Package for Plasma/Material Interactions during Plasma Instabilities

A. Hassanein and I. Konkashbaev (Troitsk Inst. for Innovation and Fusion Research)
 Journal of Nuclear Materials 273 (1999) 326-333.

An Alternative Explanation for Evidence that Xenon-depletion, Pore Formation and Grain Subdivision Start at Different Local Burnups

J. Rest and G. L. Hofman
 Journal of Nuclear Materials 277 (2000) 231-238.

DART Model for Irradiation-Induced Swelling of Uranium Silicide Dispersion Fuel Elements

J. Rest and G. L. Hofman
 Nuclear Technology, Vol. 126, pp. 88-101 (April 1999).

Behavior of Plasma-Facing Materials at Thermal Transient High Heat Fluxes

A. Hassanein
 Oral presentation at Discussion Session on Fusion Technology Issues at the 9th
 Intl. Conf. on Fusion Reactor Materials (ICFRM-9), Colorado Springs,
 Oct. 10-15, 1999.

Current Issues in Modeling Disruption Effects on Plasma Facing Components

A. Hassanein and R. Mattas
 Oral presentation at APEX Study Mtg., U. of California, Los Angeles,
 Nov. 8-11, 1999.

Effect of Plasma Instabilities on Tungsten Divertor Plate

A. Hassanein
 Oral presentation at the FIRE Physics Mtg., Princeton Plasma Physics Laboratory,
 May 1-3, 2000.

Modeling Plasma-Material Interactions in Tokamak Devices: Part II: Effects of Plasma Instabilities

A. Hassanein

Oral presentation at ALPS & APEX Joint Mtg., Argonne National Laboratory, May 8-12, 2000.

Modeling Plasma-Material Interactions in Tokamak Devices: Part I: Assessment of Particle Pumping and Removal

A. Hassanein

Oral presentation at ALPS & APEX Joint Mtg., Argonne National Laboratory, May 8-12, 2000.

Laser Electrodispersion for Fabrication of Controlled Nanostructures

A. Hassanein

Oral presentation at Fusion Power Program Mtg., Argonne National Laboratory, July 2000.

Effect of Plasma Disruptions on Liquid Surfaces

A. Hassanein

Oral presentation at APEX Electronic Mtg., Los Angeles, Aug. 24, 2000.

Erosion/Redeposition Analysis of Plasma-Facing Surfaces

A. Hassanein

Invited presentation at PFC Review Committee, Albuquerque, Dec. 8-9, 1999.

Effects of Plasma Instabilities on Plasma-Facing Components

A. Hassanein

Invited presentation at PFC Review Committee, Albuquerque, Dec. 8-9, 1999.

Fragmentation of Liquid and Solid Targets for Muon Collider Projects

A. Hassanein

Invited presentation at Neutrino Factory and Muon Collider Collaboration Meeting, Berkeley, CA, Dec. 13-15, 1999.

Recent Modeling of Target Behavior for Muon Collider and Related Projects

A. Hassanein

Invited presentation at Muon Collider Targetry Mtg., Brookhaven National Laboratory, Jan. 24-25, 2000.

Computational Modeling and Simulation Capabilities at Argonne National Laboratory

A. Hassanein

Invited presentation at Ford-Energy Technology Division Meeting, Argonne National Laboratory, March 16, 2000.

Modeling Target Behavior and Design for Muon Collider, NUMI, and Neutrino Factory

A. Hassanein

Invited presentation at Fermi National Laboratory, Jan. 27, 2000.

Liquid Metal Applications for High Power Targets

A. Hassanein

Invited presentation at Ioffe Institute, St. Petersburg, Russia, April 6, 2000.

Liquid Metal Applications for High Power Targets

A. Hassanein

Invited presentation at STC "SINTEZ," D. E. Efremov Institute, St. Petersburg, Russia, April 4, 2000.

Pulsed Heating and Hydrodynamic Effects of IFE Chamber Walls during Microexplosions

A. Hassanein

Invited presentation at ARIES Project Mtg., U. of Wisconsin, Madison, June 19-21, 2000.

GLIMMER: A Plasma-Gun Flowing Liquid Metal Target Experiment at Illinois

D. Ruzic and A. Hassanein

Oral presentation at APEX Study Mtg., U. of California, Los Angeles, Nov. 8-11, 1999.

Thermal and Electromechanics

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Principal Investigators: Y. S. Cha, S. U. Choi, K. E. Kasza, T. M. Mulcahy, N. T. Obot,
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The Thermal and Electromechanics (TEM) Section conducts research and development on new applications in thermal sciences, magnetics, electromagnetics, electrodynamics, superconductivity, and their support technologies. The work, both experimental and analytical, includes (a) developing new and/or improved correlations, prediction methods, and experimental techniques for the design and evaluation of energy-efficient and environmentally acceptable components and processes; and (b) developing and testing of components and devices.

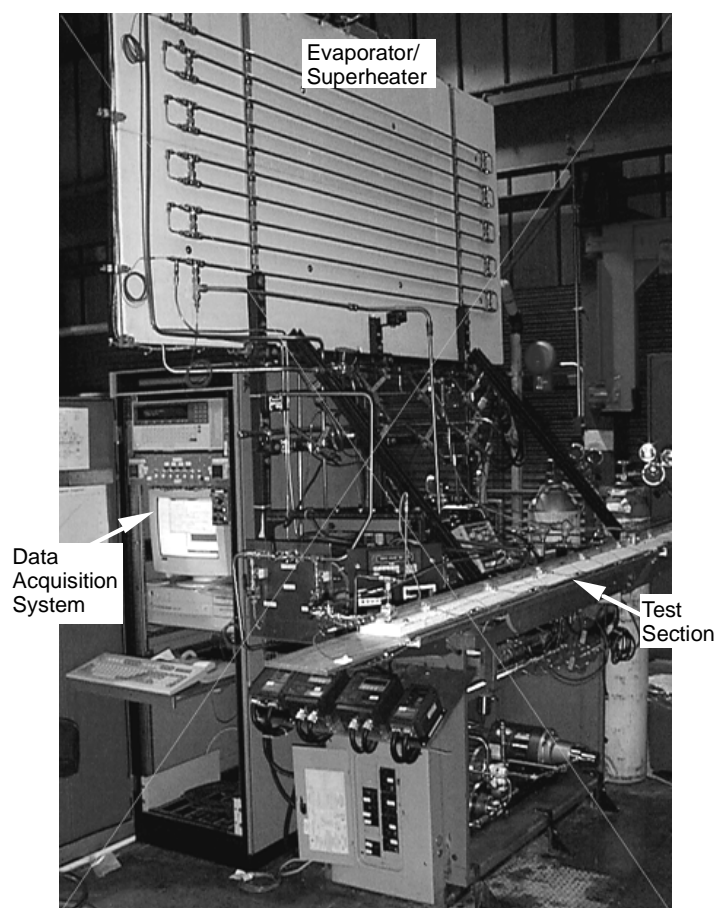
The TEM Section receives funding from several diverse sponsors, with the primary source the U.S. Department of Energy. Many projects involve collaboration with industry and have informal or formal agreements, such as cooperative R&D agreement (CRADA) relationships. Section members are thus frequently involved with industrial scientists and engineers in these collaborative programs. The Section collaborates with the ET Ceramics Section and with the Materials Science Division in the superconductivity program and with the Process Evaluation Section of the Energy Systems Division in automotive recycling. Section staff also provide support to the NRC-sponsored Steam Generator Tube Integrity Program.

Multiport Cylinder Dryer

The multiport cylinder dryer, ANL's high-performance, small-channel, heat exchanger technology for pulp and paper drying, offers a radically new approach to increasing paper-drying rates. The key feature of the new dryer design derives from the observation that the thick layer of condensate formed around the inner circumference of conventional dryers represents a major resistance to heat flow, thus severely limiting drying capacity. The basic concept of a multiport cylinder dryer uses the flow of steam through "ports," or longitudinally-oriented flow passages, close to the cylinder dryer surface. In contrast to conventional cylinder dryers, the "rim of condensate" will be minimized in the multiport cylinder dryer, and the dominant heat transfer mechanism will be forced convection, which is more effective than conduction. Furthermore, the use of multiports in new cylinder dryer designs will increase the surface area, with the result that drying rates will be significantly higher.

The Multiport Dryer project is funded by DOE's Office of Industrial Technologies to support the American Forest & Paper Association's Agenda 2020. The primary objective of the project is to develop and demonstrate a multiport drying technology that will provide the users of steam-heated cylinder dryers with increased drying rates at competitive retrofit costs.

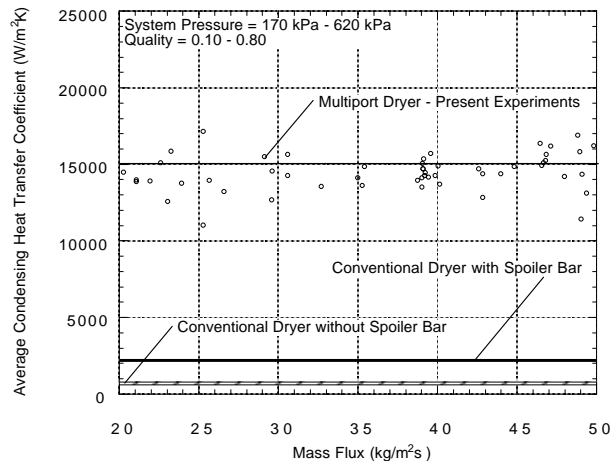
A laboratory-scale, multiport cylinder dryer heat transfer test apparatus has been designed and fabricated at ANL, in collaboration with the University of Illinois at Chicago and with Eastern Paper. The test apparatus, shown in the following figure, has been used to demonstrate the feasibility of multiport dryers for pulp and paper drying and to generate condensation heat transfer and two-phase pressure drop data. Engineers at Eastern have provided input to the basic multiport dryer design to ensure that the design is commercially viable, and to the test program, to ensure that the test conditions simulate industrial operating conditions. The Johnson Corporation has provided input on condensate removal.



Digital image of multiport dryer heat transfer test apparatus

Results of the proof-of-concept tests are very impressive in that the condensing heat transfer coefficient in a multiport channel is significantly greater than that in a conventional dryer, as shown in the next figure, and the dryer surface temperature profile is nearly uniform. Therefore, ANL's new multiport dryer technology seems to be the best approach for the pulp and paper industry's goal of doubling current evaporation rates. In fact, ANL's multiport dryer team, together with the two industrial partners, is at the frontier of drying technology R&D.

Future work includes development of design correlations, design and fabrication of a prototype multiport cylinder dryer, testing of a prototype multiport cylinder dryer in one of the research dryers at the Johnson Corporation's R&D Center, and evaluation of the full-scale test



Comparison of multiport dryers with conventional cylinder dryers. The condensing heat transfer coefficient in the multiport dryer is about seven times greater than that in a conventional dryer with spoiler bars, and about 20 times greater compared than that in a conventional dryer without spoiler bars.

results with Eastern and Johnson. With the Eastern and Johnson firms as industrial partners, the transfer of the technology to industry will be facilitated. Successful commercialization of the technology will help DOE and ANL accomplish their missions because the increased productivity will translate into significant improvements in capital effectiveness and financial performance of the country's most capital-intensive industry.

Thermal Management in Heavy Vehicles

Thermal management in heavy vehicles is cross-cutting because it directly or indirectly affects engine performance, fuel economy, safety and reliability, engine/component life, driver comfort, materials selection, emissions, maintenance, and aerodynamics. It follows that thermal management is critical to the design of large (class 6-8) trucks, especially in optimizing for energy efficiency and emissions reduction. With the trend toward more powerful engines (up to 600 hp), more air conditioning, more stringent emissions requirements, and additional auxiliary equipment, the heat rejection requirements of large trucks can be expected to grow substantially (an estimated 40-60%) in the near future. In addition, engine performance requirements are expected to increase, along with a demand for longer component life, reduced maintenance, and improved driver comfort and safety. The challenge will be to design higher-performing thermal management systems that occupy less space, are lightweight, and have reduced fluid inventory.

The Section is engaged in a study of thermal management for heavy vehicles funded by the U.S. Department of Energy, Office of Transportation Technologies, Office of Heavy Vehicle Technologies (OHVT). The work follows a roadmap developed after a Section-organized DOE workshop on truck thermal management in October 1999. The approach is to improve the efficiency of heavy vehicles from a thermal management viewpoint by investigating use of phase-change heat transfer, nanofluid coolants, evaporative cooling to enhance air-side heat

rejection, and operating the coolant at higher temperatures. Work for OHVT also includes a collaboration with Tufts University to investigate the feasibility of regenerative shock absorbers to recover energy.

Nanofluids: Fundamentals of Energy Transport and Applications in Vehicle Thermal Management Systems

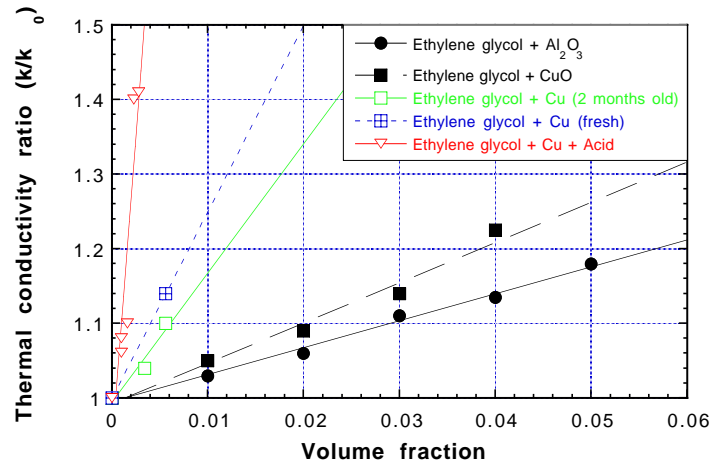
A new class of heat transfer fluid, called nanofluids, has been discovered by the Section. Nanofluids are engineered by dispersing nanometer-size solid particles in traditional heat transfer fluids to increase thermal conductivity and heat transfer performance. Experiments conducted by the Section have found that the improvement in heat-transfer properties of several nanofluids is significantly greater than that predicted by existing theory. This represents a fundamental discovery in basic heat transfer.

Nanofluids can be used to improve thermal management systems in many applications, including heavy vehicles. Also, thermal management is becoming one of the key enabling technologies leading to mass production of fuel-cell, electric, and hybrid vehicles, which must operate at temperatures significantly lower than conventional internal combustion engines. Therefore, there is a strong need to develop advanced heat transfer fluids with significantly higher thermal conductivities and improved heat transfer than are presently available and to transfer this technology to the automotive industry.

The feasibility of the nanofluid concept was originally demonstrated under an LDRD project. This work then received initial DOE support from the Office of Heavy Vehicles. More recently, it has also received support from DOE, Office of Basic Sciences. Efforts within the TEM Section have focused on measuring and characterizing the thermal and flow properties of nanofluids as a function of particle material, size, and loading. Recent research with metallic nanofluids has demonstrated that thermal conductivity can be substantially enhanced over the base fluid at very low volume fractions of nanoparticles, as shown in the figure below. As part of the effort to develop nanofluids, ANL has established a CRADA with Valvoline, Inc.

Recent research with metallic nanofluids containing <10-nm-diameter Cu nanoparticles dispersed into ethylene glycol have demonstrated that at very low volume fractions of nanoparticles, thermal conductivity can be substantially enhanced over that of the base fluid and of oxide nanofluids containing CuO or Al₂O₃ nanoparticles of average diameter 35 nm, as shown in the next figure. This is an unexpected result because established theory predicts no effect of either particle diameter or particle conductivity on nanofluid conductivity.

Because nanofluids hold great potential for improving heat transfer in many applications, a basic understanding of the thermal properties of nanofluids is essential to the systematic development of nanofluid-based heat transfer technologies. Such advances are central to the mission of DOE and will have wide-ranging economic and environmental benefits. A new project was initiated in July 2000 with the support of the DOE Office of Science, Office of Basic Energy Sciences, Division of Materials Sciences and Engineering. The objectives of this new Nanofluids Fundamentals project are to discover the key parameters that are missing from existing theories and to understand the fundamental mechanisms of thermal-conductivity enhancement of nanofluids. We have begun exploring the theoretical basis for the anomalously increased



Thermal conductivity of ethylene glycol improves with increasing volume fraction of copper oxide or aluminum oxide nanoparticles dispersed in ethylene glycol. Even greater effects are demonstrated in metallic copper nanofluids at very low volume fractions of nanoparticles.

thermal conductivity of nanofluids. One approach is to conduct molecular dynamics simulation (MDS) of nanofluids in which nanoparticle size will be considered as a dominant parameter in enhancing nanofluid thermal conductivity. Another approach is to measure the thermal conductivity of nanoparticles using the photoacoustic method. The tasks will be carried out in collaboration with Purdue University and Rensselaer Polytechnic Institute.

More recently, nanofluids containing carbon nanotubes and SiC were produced. Preliminary tests show that these new nanomaterials have the potential to become nanofluid materials of the future. Therefore, future work will focus on the production and characterization of nanofluids containing nonmetallic and other novel/exotic nanomaterials. Experimental data will be used to validate MDS of nanofluids. Also, we will work on the heat transfer and flow behavior of nanofluids, and we will evaluate the friction and wear properties of materials in contact with the nanofluids for application in thermal management systems.

Critical Heat Flux

Currently, cooling systems in heavy vehicles are designed to use a 50/50 mixture of ethylene glycol and water in the liquid state. The heat sink is ambient air, and most of the heat is transferred in the radiator. The amount of heat rejected in the radiator is limited by current radiator designs that are essentially optimal. In addition, precision cooling with the 50/50 liquid mixture is limited by geometry and liquid properties. One method to increase heat transfer rates for both radiator and precision cooling applications is to design the cooling systems to operate with a boiling fluid under certain conditions and/or in certain areas of the engine.

Order-of-magnitude-higher heat transfer rates can be achieved in nucleate-boiling cooling systems than in conventional, single-phase, forced-convective cooling systems. However, boiling of the coolant in an engine, by design or by circumstance, is limited by the

critical heat flux phenomenon. The objectives of this project are to (a) verify the feasibility of nucleate-boiling cooling systems; (b) experimentally investigate the characteristics of coolant boiling and critical heat flux (CHF) under conditions of horizontal flow, small channel, and low mass flux; and (c) develop predictive methods for boiling heat transfer and CHF under engine application conditions.

This project, funded by the U.S. Department of Energy, Office of Heavy Vehicle Technologies and Office of Advanced Automotive Technologies and in collaboration with the University of Illinois at Chicago. A CHF Test Apparatus (see next figure) has been designed and fabricated at ANL to study boiling heat transfer and CHF of flowing water, ethylene glycol, and aqueous mixtures of ethylene glycol under heavy-vehicle-engine conditions. Preliminary experiments were performed with water at a system pressure of 203 kPa (saturation temperature = 120.5°C), mass fluxes of 50-200 kg/m²s, and inlet temperatures from ambient to 80°C. The experimental data are in good agreement with the state-of-art boiling heat transfer correlation of the Chen and modified small-channel boiling heat transfer correlation of ANL. The results of CHF experiments show that CHF occurs in the range of high qualities from 0.7 to 1.0, which indicates that the nucleate-boiling cooling system can remove very high levels of heat from the engine because of coolant boiling before the occurrence of CHF.

The CHF Test Apparatus has recently been reconfigured to allow measurement of the component fraction of both the liquid and gas phases by means of a refractometer and a residual gas analyzer. In addition to the 50/50 ethylene glycol/water mixtures, future work will investigate higher boiling point fluids, such as pure propylene glycol and ethylene glycol.



Critical-heat-flux test apparatus without thermal insulation

Small-Channel Evaporator

Only a few studies in the literature report on two-phase fluid flow and heat transfer in compact heat exchangers. Nevertheless, extensive applications exist in the process industries, where phase-change heat transfer allows more compact heat exchanger designs with better performance than those used for single-phase operation. To further the application of compact heat exchangers in the process industries, there is a need to understand the fundamental issues of two-phase flow and heat transfer in small channels representative of compact heat exchanger flow passages.

Two-phase-flow pressure drop measurements were made as part of flow-boiling heat transfer studies funded by the DOE Office of Energy Efficiency and Renewable Energy and the Office of Science. Testing was performed with three refrigerants (R-134a, R-12, and R-113) at six different pressures ranging from 138 to 856 kPa, and in two sizes of round tubes (2.46 and 2.92 mm inside diameters) and in one rectangular channel (4.06 x 1.7 mm). State-of-the-art large-tube correlations failed to satisfactorily predict the experimental data. A new correlation for two-phase flow frictional pressure drop in small channels was developed, taking into account the effects of surface tension and channel size; it is applicable for smooth tubes with hydraulic diameters of ≈ 3 mm for the three refrigerants tested. The correlation was tested against the experimental data for the three refrigerants; the error was $\pm 20\%$.

Ice Slurries for District Cooling

ANL, under U.S. DOE funding, pioneered the development and fostering of ice slurries for district cooling. The cooling capability of an ice slurry pumped to a load can be up to five times greater than that associated with chilled water delivered at the same mass flow rate. This high cooling capacity also allows slurry transmission piping and storage tank sizes to be reduced significantly over that required with conventional, chilled-water cooling systems. Today, both domestic and foreign interests are considering the adoption of this technology for cooling large loads.

The storage of ice particulate slurry in tanks, however, is complicated because under certain not well understood conditions, the ice crystals will progressively grow together, or agglomerate, making pumping of the slurry from the tank very difficult. It is desirable to store the slurry at the highest packing density possible in order to most effectively utilize the available tank size. Therefore, even if the ice does not agglomerate in the tank, it must be carefully diluted to a packing density that is compatible with pumping it through a pipe delivery network. No reliable means has been developed for tank-slurry extraction or ice-loading control. In a two-year research project begun in July 1997 in collaboration with NKK Corporation of Japan, the ANL Ice Slurry Test Facility has been reconfigured and rebuilt (see next figure), and is being used in the performance of experimental studies on the factors influencing ice crystal agglomeration in ice slurry storage tanks. These studies are also developing methods for minimizing agglomeration and for improving the efficiency and controllability of tank extraction of slurry for distribution to cooling loads.

In a study of the factors affecting ice particle agglomeration during storage, instrumentation for monitoring the change in ice bed packing conditions based on local

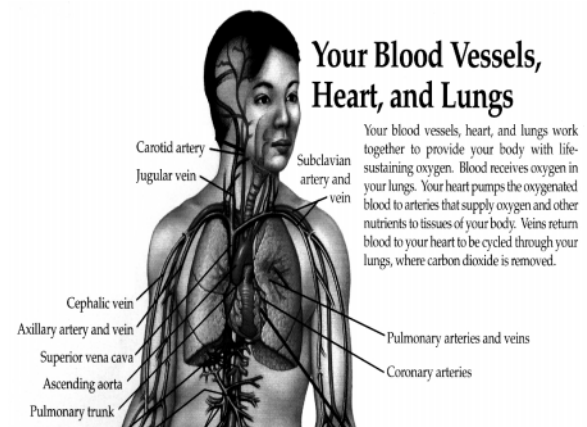


ANL Ice Slurry Test Facility

resistivity measurement has been developed and a patent application has been filed. We also discovered that adding <1% of a freezing-point depressant to the stored slurry can suppress slurry agglomeration for several days and greatly facilitate extraction of the slurry from storage for distribution to cooling loads. The ongoing studies are addressing the remaining engineering issues that are impediments to the utilization of the ice-slurry cooling concept.

Using Targeted Ice Slurry Cooling to Induce Protective Heart and Brain Hypothermia for Medical Treatment of Cardiac Arrest/Stroke

The Section has pioneered the use of ice slurries in a unique biomedical application. Currently, survival from out-of-hospital cardiac arrest is poor, since cells in the brain and heart begin to die within minutes. Similar difficulties also pertain to stroke patients. Many believe the ability of cells to survive ischemia could be significantly enhanced by rapid cooling of the heart and brain, i.e., by resuscitative hypothermia. Despite the potential benefits, few studies have addressed the heat transfer difficulties associated with attempting resuscitative hypothermia without bypass procedures. The Section is collaborating with the University of Chicago Medical School's Department of Emergency Medicine to investigate the use of ice slurries in



The carotid artery, jugular vein, and lungs are used as in-body ice slurry heat exchangers to achieve rapid targeted cooling of the heart and brain

this process. Initial results indicate that rapid induction of protective hypothermia is feasible in the out-of-hospital environment without the use of complex time-consuming bypass external heat exchangers by using in-body biological heat exchangers cooled with ice particle slurry.

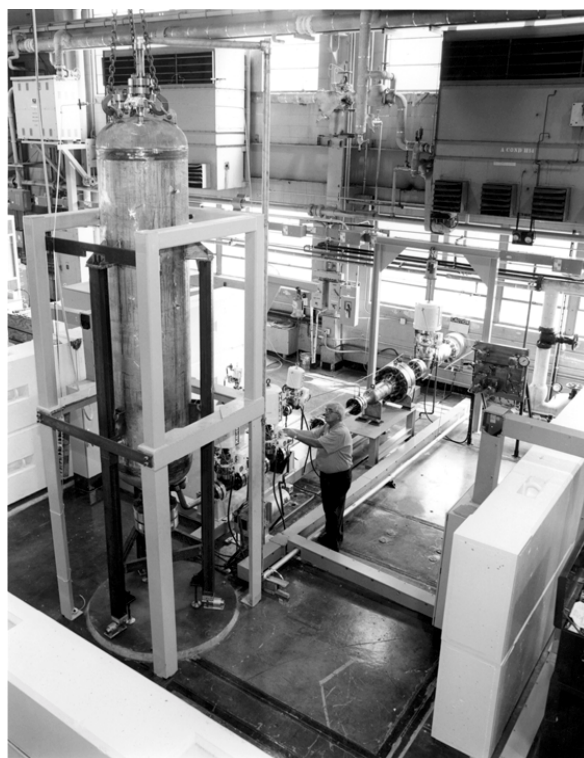
Ice particle slurries can have up to 10 times the cooling capacity of single-phase coolants such as chilled saline. Recent ANL modeling and analysis of brain and heart cooling has shown that external cooling alone cannot achieve the desired fast rate of cooling and suggest that ice slurry cooling of the target areas would be very effective. Furthermore, recent laboratory experiments in ANL's Ice Slurry Research Facility have demonstrated that ice particle phase-change slurries can be engineered with chemicals that are compatible with human tissue and have the desired fluidity. The medical use of slurries for cooling has its technical foundations in ANL's development of ice slurry cooling for buildings. The medical use of slurry is unique and involves many new engineering considerations, including entirely new types of slurry constituents and new methods of production and delivery.

ANL's Ice Slurry Research has developed slurries that are compatible with human tissue and have the needed fluidity to be administered through small-diameter tubing and hypodermic needles. Swine tests using ANL-engineered slurry at the University of Chicago produced heart and brain cooling rates 20 times greater than conventional methods. ANL and the University of Chicago filed three patent applications on making and using slurry for rapidly cooling the heart and brain. Intellectual property licensing rights negotiations are underway with a private company. A proposal for further development of this technique has been submitted to NIH.

Nuclear Steam Generator Tube Integrity

The Energy Technology Division is conducting for the NRC a program on nuclear steam generator tube integrity. A Section staff engineer is participating in the program as lead engineer for the design, fabrication, and operation of two new, large-scale facilities for generating unique data supporting an improved understanding of the nature of stress-corrosion-cracked (SCC) flawed steam generator tube leakage flows and rupture characteristics.

The overall objective of this program is to provide the experimental data and predictive correlations and models needed to permit the NRC to independently evaluate the integrity of steam generator tubes as plants age and degradation proceeds, new forms of degradation appear, and new defect-specific management schemes are



ANL's steam generator tube pressure and leak-rate blowdown test facility

implemented. Operation of these facilities have generated new unique insight into the complex behavior of flawed steam generator tubes.

The facilities include a High-Temperature Pressure and Leak-Rate Blowdown Facility (temperatures up to 343°C, pressures of up to 21 MPa, and pressurized-water flow rates up to 1520 L/min) for conducting tube failure and leak-rate tests under simulated prototype steam generator operating conditions. The Room-Temperature High-Pressure Facility (pressures up to 52 MPa and flow rates up to 48.4 L/min) is capable of testing at pressures associated with mandated overpressure safety margins.

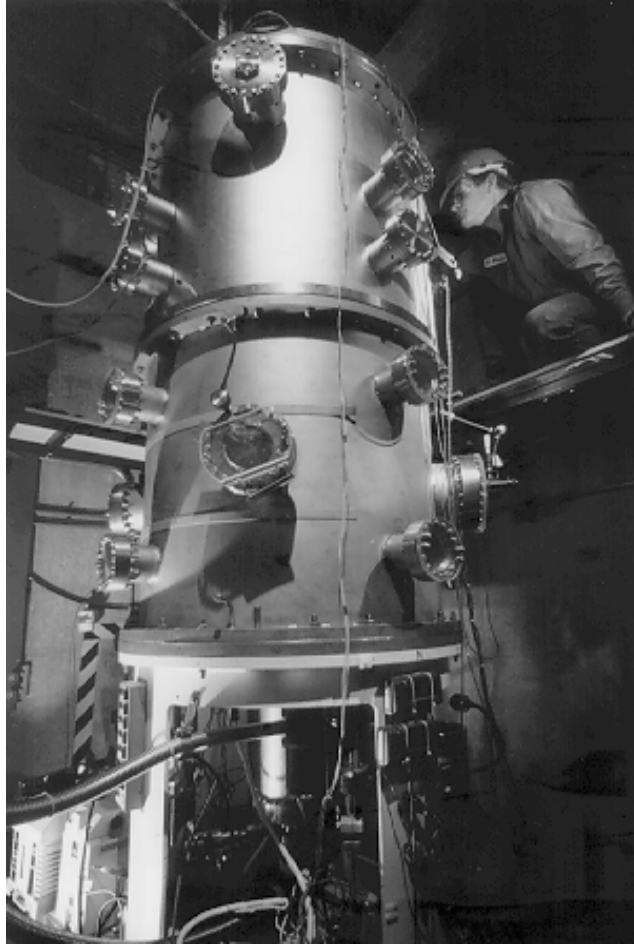
Flywheel Energy Storage

Past work in the Section explored the use of high-temperature superconductors (HTSs) in magnetic bearing applications, and a number of techniques were developed in order to produce an HTS bearing with extremely low rotational loss. We have demonstrated such a bearing with a rotational drag more than two orders of magnitude lower than that of a conventional magnetic bearing and several orders of magnitude lower than that of mechanical bearings. With this bearing, an energy-efficient flywheel could be constructed with a bearing loss of <2%/day (including parasitic power to cool the HTS). Such a flywheel has potential for use in diurnal utility energy storage or as a replacement for batteries in an electric vehicle. Several patents on this bearing/flywheel concept have been granted and several more are in process. The HTS bearing work, as a result of substantial funding from and collaboration with Commonwealth Research Corporation, has evolved into a flywheel energy-storage program.

An important test apparatus in this program is a glass bell-jar vacuum chamber with an oil-diffusion pump. This apparatus is capable of testing bearings with rotating masses of up to ≈ 1 kg. It has been used to test an Evershed-type HTS bearing, i.e., an HTS bearing in which most of the levitation is accomplished by an attractive force between a stationary permanent magnet and the permanent magnet rotor, while the HTS is used to provide stiffness and stability. A coefficient of friction of 10^{-8} has been obtained with this bearing. Experiments with this type of bearing have shown that velocity-dependent losses in such systems can be almost completely eliminated. Another novel HTS bearing, called the mixed- μ bearing, was tested in this apparatus and demonstrated coefficients of friction of 10^{-9} , two orders of magnitude lower than the conventional HTS bearing.

A second apparatus, shown in the next figure, is a stainless steel vacuum chamber equipped with a turbomolecular vacuum pump and that is capable of testing rotating masses of >100 kg. A steel-reinforced concrete pit has been modified to safely test flywheels with rotational energies of 10 kWh and higher. We have spun 10- and 170-kg rotors to 20,000 rpm in this highly instrumented chamber, which can be readily adapted to study the performance of various flywheel sizes and configurations.

A third vacuum-chamber apparatus is a burst chamber, in which we use HTS bearings to spin small rotors to destruction. These experiments allow us to find the failure modes for various flywheel designs, help determine the maximum safe speed for our larger rotors, and aid in validating various stress models used to analyze the flywheel rotors. The chamber has also been used to test larger components at rotational speed before incorporating them into large flywheels.



This stainless steel vacuum chamber can be used to test rotating masses of more than 100 kg at very high rotational speeds

A fourth apparatus presently uses part of the burst chamber to form a stand-alone flywheel unit. This apparatus has a heavy steel shell capable of confining a failure of a 5-kWh flywheel. The intent of this apparatus is to form a fieldable flywheel energy storage unit that can be tested on the utility grid. This apparatus is shown in the next figure.

In a separate project, the Section is collaborating with Boeing to develop a flywheel energy storage device. ET's portion of this project is funded by DOE. In this program, we have shown that a simple design tool, using the concept of "frozen images," can be used to predict the levitation force and bearing stiffnesses of most HTS bearing systems. This design method speeds up the design process by more than one order of magnitude. We have also identified several motor/generator concepts that are compatible with HTS bearings. Initial analysis and experiments showed that a Halbach-array internal-dipole motor/generator had very low stiffness and operated well with HTS bearings. More recently, we have shown that a cup-motor concept with rotating backiron has a higher specific power than the Halbach and has a sufficiently low stiffness to be useful with flywheels using HTS bearings.

Fault-Current Limiter

In a 1998 agreement, S & C Electric (a leading U.S. manufacturer of high-voltage switching and protection devices for electric power industry) and ANL jointly carried out a Phase I feasibility study on a passive superconducting fault-current-limiter (FCL) concept known as the superconductor shielded core reactor (SSCR). In this study, S & C identified three specific applications and provided electrical specification for the FCL. ANL has designed and constructed a scaled model (480 V/60 A) of an SSCR that uses a melt-cast processed BSCCO-2212 superconductor tube. The SSCR was tested extensively at S & C's high-voltage laboratory. The major findings of the joint program include: (a) a closed-core SSCR behaves like a mostly resistive device with relatively large limiting capability and dissipation, (b) an open-core SSCR behaves like a hybrid inductive/resistive device with reduced limiting capability and dissipation, and (c) both the closed-core and the open-core SSCR can limit the fault current before the first current peak. While the melt-cast processed BSCCO-2212 tube appears to be working, there is evidence that the newly emerging melt-textured bulk YBCO may have superior properties (such as critical current density and mechanical strength). Furthermore, we expect that in the near future, relatively large melt-textured YBCO rings can be manufactured at relatively low cost (comparable to that of melt-cast processed BSCCO-2212 tube). S & C would like to test the performance of the YBCO-based SSCR in the next phase of the program, which is expected to begin during FY 2001. The major task will be (a) to characterize the electromagnetic properties of the large YBCO rings, (b) measure the performance of the YBCO-ring-based FCL, and (c) compare the performance of the YBCO-ring-based FCL with that of the BSCCO-tube based FCL.



Energy storage unit sized for testing on a utility grid to 5 kWh

Permanent Magnet Fabrication

Another Section project, funded by DOE's Office of Advanced Automotive Technologies (OAAT), is improving the production process for low-cost, high-energy-product permanent magnets. Emphasis is being placed on reducing the weight of vehicles. Electric motors are in use in many vehicle systems and offer a significant opportunity for weight reduction. If traction motors are used for electric drive, the opportunity for weight reduction will be even greater. One of the most promising ways to reduce weight in an electric motor is by increasing the energy product of the permanent magnets used in the motors. A higher energy product in the permanent magnet will not only reduce the weight of the permanent magnet, but will also reduce by a similar fraction the weight in the balance of the motor in obtaining the same

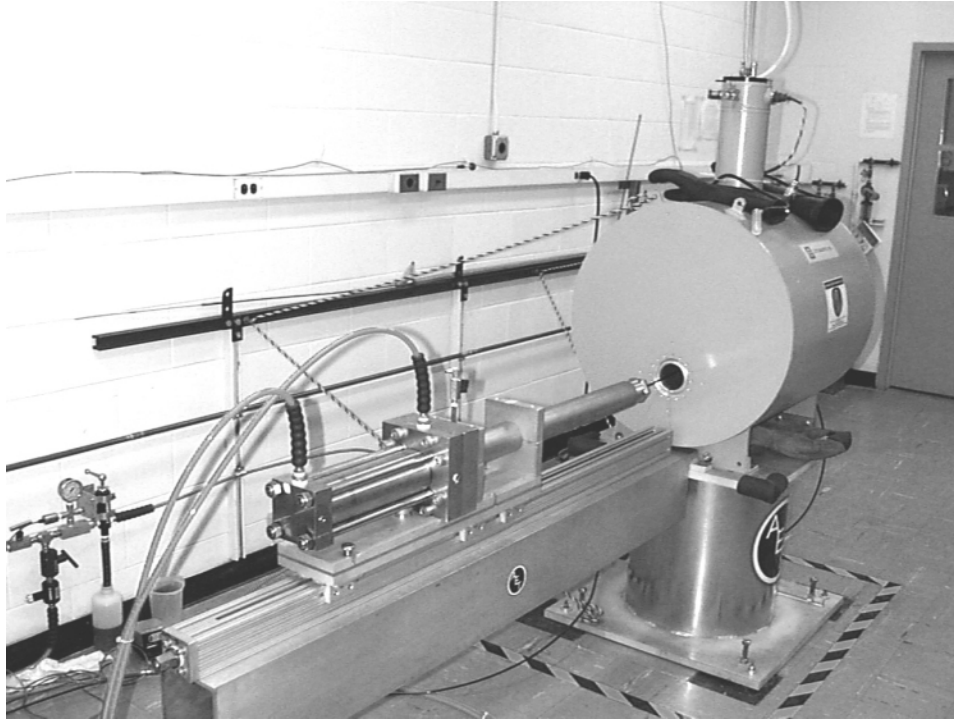
torque. Energy products in permanent magnets are currently limited by manufacturing processes and are far below the theoretical potential. A further barrier to a more widespread use of these permanent magnets in vehicles is their cost of production. The same process that we have proposed for increasing the energy product is also likely to significantly decrease the manufacturing cost.

The remanent magnetic field is one of the factors in the energy product of a permanent magnet and can be increased by improving the alignment of the magnetic domains before the permanent magnet is sintered. One method to accomplish this is with higher magnetic fields during the pressing process. These magnetic fields are limited in present manufacturing processes by the cooling and structural requirements of pulsed electromagnets. To obtain higher magnetic fields, the use of superconducting magnets will be required. The use of a superconducting magnet will eliminate the joule-heating costs in the pressing operation, but the cost of electricity (probably several cents per kg) is a very small fraction of the total cost in manufacturing a permanent magnet. Much greater savings would result from a better uniformity of orientation in the final product. When the particles are aligned in the magnetic field, they tend to follow the magnetic field lines. These lines are distorted by the self-demagnetizing factor of the magnetized particles, i.e., the magnetic field lines generated by the piece being fabricated. With an external magnetizing field of 3-4 T, a self-demagnetizing field of even 1 T is a significant distortion. If a magnetizing field of 8 T or higher were used, this self-demagnetizing field becomes a smaller fraction of the total, and the block being magnetized becomes more uniform. In terms of the manufacturing process, the yield of high-energy-product pieces from a magnetized block would increase significantly, and thus the cost of the manufactured permanent magnet should significantly decrease.

Permanent-magnet manufacturers do not presently use superconducting magnets because they do not cycle quickly. A reciprocating feed will allow the use of superconducting magnets that need not cycle at a production rate equal to that of the present system using electromagnets. We have constructed a large-bore 8-T magnet and associated powder press (see next figure). We have already pressed NdFeB powder that after sintering has a higher energy product than that obtainable by manufacturers with conventional techniques. We are working with several major U.S. manufacturers of NdFeB permanent magnets to optimize the production process with their powders.

Recycling of Future Vehicles

Another Section project involves development of technologies for cost-effective recycling of Advanced Automotive Technologies (AAT). This project is part of a larger Auto Recycling program initiative at ANL and involves collaborative efforts with the ES Division's Process Evaluation Section and other R&D groups. Efforts in the Section to date have focused on development of an AAT Recycling R&D Program Plan for OAAT that will establish priority recycling needs and contribute toward achievement of PNGV and other AAT goals.



8-T superconducting magnet and axial press for fabrication of permanent magnets with high energy products

The automobile of the future will use significantly greater amounts of lightweight materials and more sophisticated and complex components in order to achieve fuel economy goals of 80 mpg by 2004 and 100 mpg by 2011 for baseline six-passenger sedans. The Recycling R&D Plan development effort involves identification of critical recycling needs to accomplish the ambitious goal of recycling 85% of the materials used in these high-efficiency, low-emission, vehicles, relative to 75% recyclability achieved by today's recycling industry infrastructure. This provides an R&D challenge because PNGV/AAT vehicles will utilize new lightweight materials and sophisticated components, many of which have never been processed by the automotive recycling industry. In addition, the inevitably higher purchase costs of PNGV technologies, relative to those of conventional cars, requires enhanced recyclability in order to achieve life-cycle cost effectiveness and to maintain market competitiveness.

Recent Work by the Thermal and Electromechanics Section

Publications

Magnetic Diffusion in High- T_C Superconductors

Y. S. Cha

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Mechanisms of Heat Flow in Suspensions of Nano-Sized Particles (Nanofluids)

P. Keblinski (Rensselaer Polytechnic Institute), S. Phillpot, S. U. Choi,
and J. A. Eastman

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F. E. Lockwood, G. Z. Zhang (Valvoline Co.); S. U. Choi, and W. Yu

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K. L. Uherka

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J. R. Hull

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J. R. Hull

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Sensors, Instrumentation, and Nondestructive Evaluation

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The Sensors, Instrumentation, and Nondestructive Evaluation (SI&NDE) Section conducts research and development related to instruments and NDE techniques for characterization of materials and system parameter determination for energy systems under development for several DOE offices, including Fossil Energy (FE), Energy Efficiency and Renewable (EE), Environmental Management (EM), and nuclear technologies. However, the Section also develops sensors and technologies for non-energy-producing technologies such as DOE's Nonproliferation and National Security Programs and the Nuclear Regulatory Commission (NRC). The Section also utilizes its sensors and NDE development capabilities for national initiatives such as the Partnership for a New Generation of Vehicles (PNGV), Manufacturing, Industries of the Future (IOF), and others related to the general competitiveness of American industry. In the past two years, the Section has become involved in biomedical programs for the National Institutes of Health and DOE. The projects described below give a clear indication of the breadth of our work.

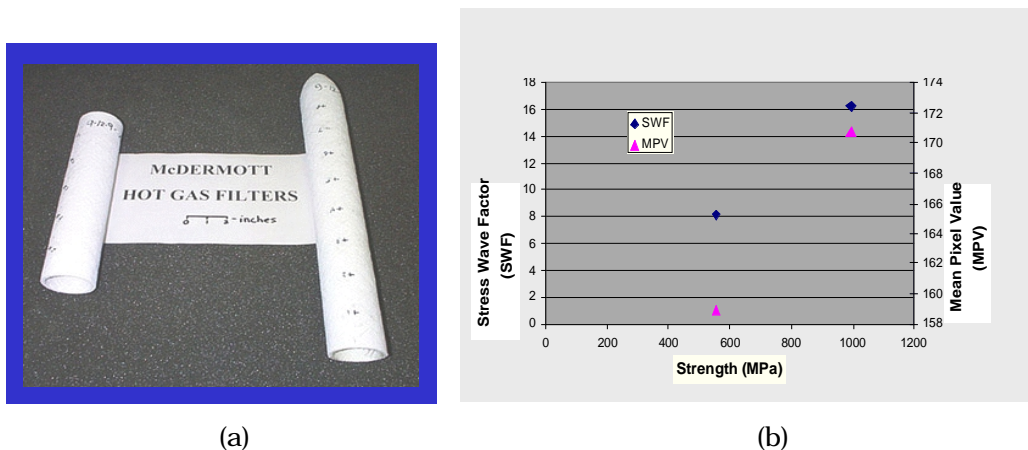
DOE Office of Fossil Energy

Development of Nondestructive Characterization Methods for Ceramic Materials

This work is part of the overall materials technology development effort within the Fossil Energy Advanced Research and Technology Development (AR&TD) area. The focus is on developing nondestructive evaluation (NDE) methods for ceramic materials utilized in fossil energy systems. Of current technological interest are (a) rigid ceramic hot-gas filters for hot gas stream cleanup (both monolithics and composites), (b) thermal barrier coatings for high-temperature sections of gas turbines, and (c) ceramics for high-temperature H₂ and O₂ separation membranes for H₂ and O₂ production from coal combustion.

We are developing several NDE methods to address advanced application of ceramic materials systems in fossil energy systems. First, we are working on acousto-ultrasonic methods for determining damage in hot-gas filters as a function of axial position along the length of the filters. Both monolithic and oxide composite materials are under study. Work to

date has demonstrated that this NDE method can be directly correlated to remaining strength of the hot-gas filter material. This is shown in the figure below for oxide composites. Cooperation on this project is extensive and includes (a) the University of Tampere in Finland, Southern Research Institute, Southern Company Services, and Oak Ridge National Laboratory.

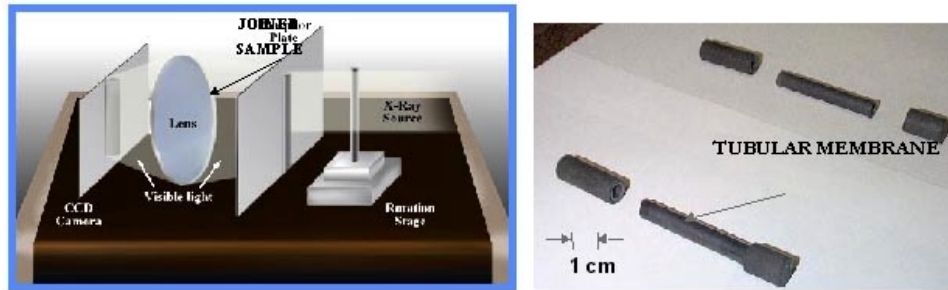


Oxide/oxide porous hot gas filters: (a) photo of two oxide test samples; (b) correlation of NDE data with strength

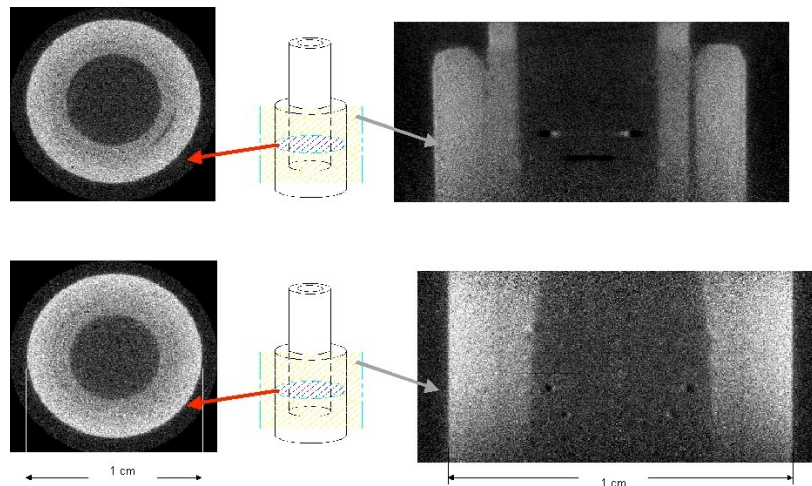
Recent advances in iron aluminides for hot-gas filters have also been made. The ANL acousto-ultrasound method was recently demonstrated to work on these materials. For cost-effectiveness, an automated system is under construction with a target of characterizing a full-length 1.5-m filter in less than 5 minutes. High-temperature oxygen and hydrogen separators are necessary to allow oxygen and hydrogen production from coal combustion. These separation membranes must be of high uniformity and structural integrity. As production of these membranes comes closer to reality, NDE methods are necessary to ensure uniformity and product reliability. For this effort, use has been made of the microimaging station at ANL's Advanced Photon Source (APS). In cooperation with The University of Chicago, sections of various H₂ separation membranes have been studied. The primary emphasis is on the joints necessary to make the H and O separation membranes feasible. Results from the APS microimaging station clearly show that this method has the spatial resolution necessary for these types of analyses. The next figure shows the microimaging station and results from the synchrotron imaging. Clear detection of leaking joints can be seen.

DOE Office of Transportation Technologies

There are two separate aspects to the NDE work being done under the OTT Office of Heavy-Vehicle Technology Materials. The first is development of NDE technology for valve train components, and the second is NDE technology development for fuel delivery and insulating components.



(a)



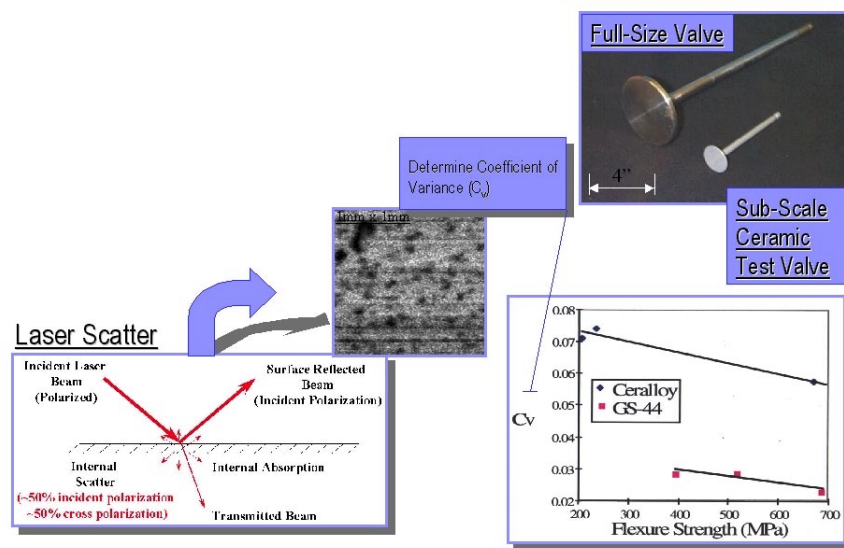
(b)

Use of synchrotron micro X-ray imaging to characterize high-temperature ceramic membranes: (a) schematic of illustration setup; (b) images showing leaking and nonleaking joints (upper and lower, respectively)

NDE Development for Valve Train components

As part of the DOE Heavy Vehicle Technology Materials Program, ceramic valves are being developed. This is a cooperative program with Caterpillar Technical Center in Peoria, IL, and has two separate parts. The first part involves study of NDE methods to detect and quantify machining-induced damage on valve seats for aggressively machined ceramic valves. The goal is to develop ANL's elastic optical scattering method for application to machining-damage detection in aggressive machining conditions designed to reduce this important cost element of ceramic valve production lasers in the visible range, e.g., He-Ne at 0.632 μm data and flexural strength of the materials. Speed has been increased to the point where the mechanical motion speed of the mechanical stages is now the physical limit. The next figure contains a schematic diagram of the method, a photo of valves, and a correlation of NDE data with strength.

The second involves NDE for ceramic-metal joints under development by Caterpillar to reduce costs for this valve train component. This work is aimed at developing NDE technologies for defect detection in ceramic-metal joints. Microstructural analysis has found cracking in



NDE technology for diesel-engine ceramic valves

joint interfaces that may significantly reduce the strength of the joint. NDE techniques under development for detection and characterization of cracks in joints include impact acoustic resonance, thermal imaging, immersion ultrasonic scanning, and X-ray CT imaging.

Advanced Sensor Technology for Next-Generation Vehicles

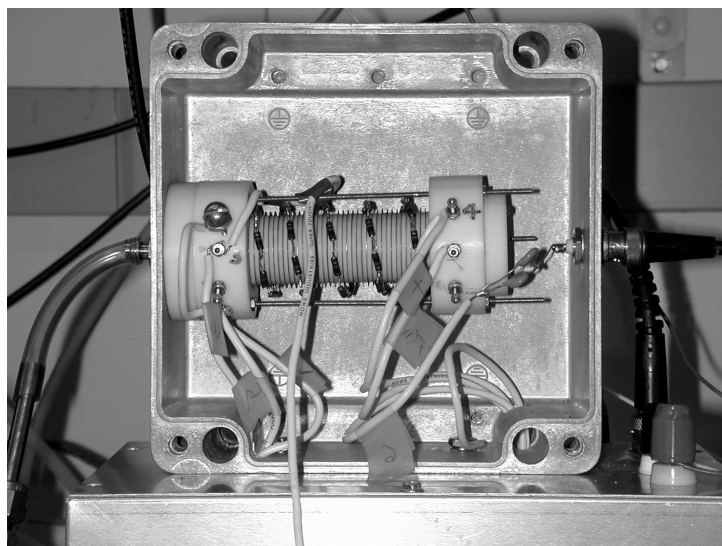
This is a CRADA project with the U.S. Council on Automobile Research (USCAR). The objective of this project is to develop an ion-mobility sensor (ISM) system capable of monitoring engine-out emissions from an internal combustion engine.

In FY 2000, we completed a series of prototype IMS tests at GM's sensor transient response reactor facility. The purpose was to demonstrate IMS capability for detecting NO_2 . Results with a radioactive Ni-63 ion source show that sensor response time is about 40 ms, which satisfies the ultimate sensor requirement. The results also indicate that by monitoring the mobility peak position of the negative NO_2 ions, the IMS can provide a quantitative measure of NO_2 concentration in both lean and rich feeds. Some difficulties were also identified:

- Lack of sensitivity in detecting NO.
- Interference due to water vapor and oxygen gas.
- Nonlinear dependence (step change in peak position at low concentrations of NO_2).
- Memory effects (relatively long cleanup time).
- Flow rate effect (poor signal-to-noise for flow rates >200 sccm).

The project later focused on development of a nonradioactive ion source because the radioactive source is generally not acceptable to the auto industry. A corona discharge

technique was proposed, and a feasibility test was conducted with a needle electrode. Evaluation of various design parameters of an NO₂-detecting ion source based on corona discharge is continuing. The figure below shows a laboratory prototype of the IMS sensor.



Ion mobility sensor

Acoustic Fuel Vapor Sensor

This was a new project starting in FY 2000. The objective is to develop low-cost, fast-response, acoustic-based fuel vapor sensors that can be used in the harsh automotive engine environment to measure or monitor fuel vapor mass flow rate and to detect variations in fuel-vapor composition. This project is a joint effort of ANL, Ford Motor Company, and Northwestern University (NU).

Vehicles sold in the U.S. use a carbon canister to trap fuel vapors from the fuel tank. The fuel vapors are purged from the canister into the engine where they are burned. The present control strategy uses the oxygen sensor in the exhaust gas manifold to correct rich shifts in air-to-fuel ratio caused by the additional fuel vapors added to the engine during the canister purge. The new requirement to trap all fuel vapors during refueling increases the amount of fuel vapors to be purged into the engine during a drive cycle. These high canister-purge flow rates make it more difficult to reduce or eliminate driveability and stall problems and to minimize disturbances in air-to-fuel ratio, possibly creating an even bigger challenge in meeting the increasingly stringent tailpipe emissions requirements. A feed-forward system, in which a fuel vapor sensor in the canister purge line would measure the mass of fuel vapors, is expected to result in a much more robust canister purge system. This would result in lower tailpipe emissions, fewer driveability problems, and a modest improvement in fuel economy by better controlling the air-to-fuel ratio. However, no such automotive fuel vapor sensor currently exists. The next figure shows a laboratory test cell used for fuel-vapor sensor development.

ANL will conduct sensor development based on speed-of-sound (SoS) and acoustic relaxation spectroscopy (ARS) measurements. The ARS measurement will be verified by a theoretical model to be developed by NU. Both SoS and ARS results will be correlated with fuel-vapor composition and concentration. The fuel-vapor sensor will then be tested at Ford.



Fuel vapor sensor

NDE Development for Fuel Delivery and Insulating Materials

The second part of our work for the Office of Heavy Vehicles/Materials Technology effort is focused on the critical issue of fuel delivery and insulating materials. In diesel engines, fuel injectors control many aspects of combustion and hence emissions. Because wear of high-pressure fuel injector pins causes poor combustion and high emissions, new ceramic fuel pins are under development. Subsurface cracks, damage induced by machining, and other defects are of critical importance. ANL is developing laser scattering methods to study the as-machined surfaces of these critical plungers. In addition, use of femto-second lasers is under study as a low-cost machining method for the critical holes. ANL is developing NDE methods to characterize the laser-drilled holes. This part of our work is a cooperative effort with Cummins Engine, Inc. Reduction of heat losses from the combustion chambers of diesel engines is still a concern. Almost always the best reduction can be achieved by using ceramic components as linings for the chamber, but low cost and high reliability remain the primary challenges here. ANL is working with Caterpillar to develop cost-effective and reliable ceramic cylinder lining components. The main effort at ANL is on nondestructive characterization of as-produced ceramic liners.

DOE Office Of Power Technology

NDE Technology Development for Microturbines

Development of microturbines to drive electric generators, that is, natural-gas-fired turbines which produce power in the range of 25-300 kWe, is a DOE initiative that began in FY 1999. Development of these small turbines is part of the larger DOE effort on efficient low-emission distributed-energy resources. The microturbines can also be combined with fuel cells or batteries for very efficient hybrid transportation power systems. Because the current microturbines have relatively low operating efficiency (usually less than 30%), a current DOE goal is to increase the efficiency to more than 40%, beginning with an increase in the hot-section temperature. High-performance monolithic structural ceramics, if produced cheaply enough, and with high enough reliability, are under development as one step in achieving the higher efficiency. ANL's role is to develop a complete volumetric, cost-effective, nondestructive,

production-line characterization method (see next figure). The ceramic rotor production target is to produce 500-1000 fully operational rotors per month. Under development at ANL is a high-speed 3D X-ray tomographic imaging system that uses new amorphous silicon X-ray area detectors. Up to 17 by 17 inches in size and with 14-bit dynamic range, these new detectors have up to 1024 x 1024 individual pixels, each 400 by 400 μm . Readout speed of the entire detector is currently 4-8 frames per second. ANL's target is to reconstruct a total volume of size to meet microturbine wheel needs in less than 10 minutes. Reaching this high rate of data acquisition and image reconstruction requires that we also investigate the use of massively parallelized computers such as the Beowulf configuration; we are pursuing this. The 3D X-ray imaging effort is being done in cooperation with Honeywell Engines and Systems, Ceramic Components Division. In addition, research test results have shown that even though the Si_3N_4 ceramic materials will allow higher hot-section temperatures to be reached, the materials degrade too fast and thus new steps are being undertaken to develop environmental barrier coatings (EBCs). These coatings must be light in weight because a high mass would cause too high a rotational load and thus a higher likelihood of failure. ANL's role in EBC development is to develop full-field scanning NDE methods to characterize, during engine shutdowns, the EBC coatings for adhesion, thickness uniformity, incomplete coverage (e.g., pinholes), and possible spalled regions. For this work, ANL is utilizing its patented elastic optical scattering method with low-power lasers, in cooperation with others involved in microturbine materials development, including Oak Ridge National Laboratory, Honeywell Engines and Systems, and United Technologies.

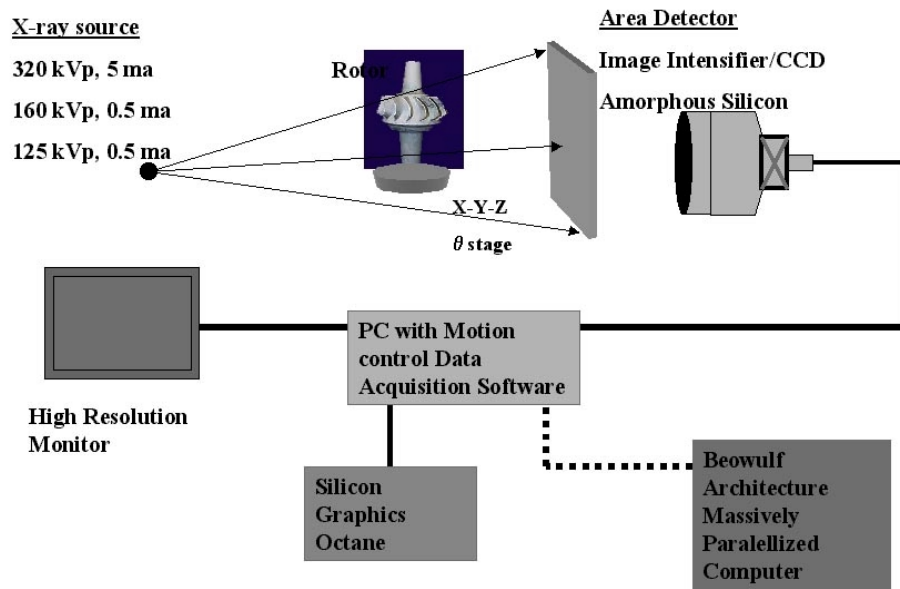
NDE Development for Ceramics in Stationary Gas Turbines

As part of the DOE Office of Power Technology Program on Advanced Turbine Systems (ATS), ANL is a subcontractor to Solar Turbines, Inc. (a wholly owned subsidiary of Caterpillar Inc.), which is developing and evaluating ceramics for application to stationary gas turbines in a program titled "Ceramics in Stationary Gas Turbines." The ANL effort is directed toward developing NDE methods for these ceramic materials that are to be used in the Centaur H and Mercury gas turbine engines intended for stationary electric power generation.

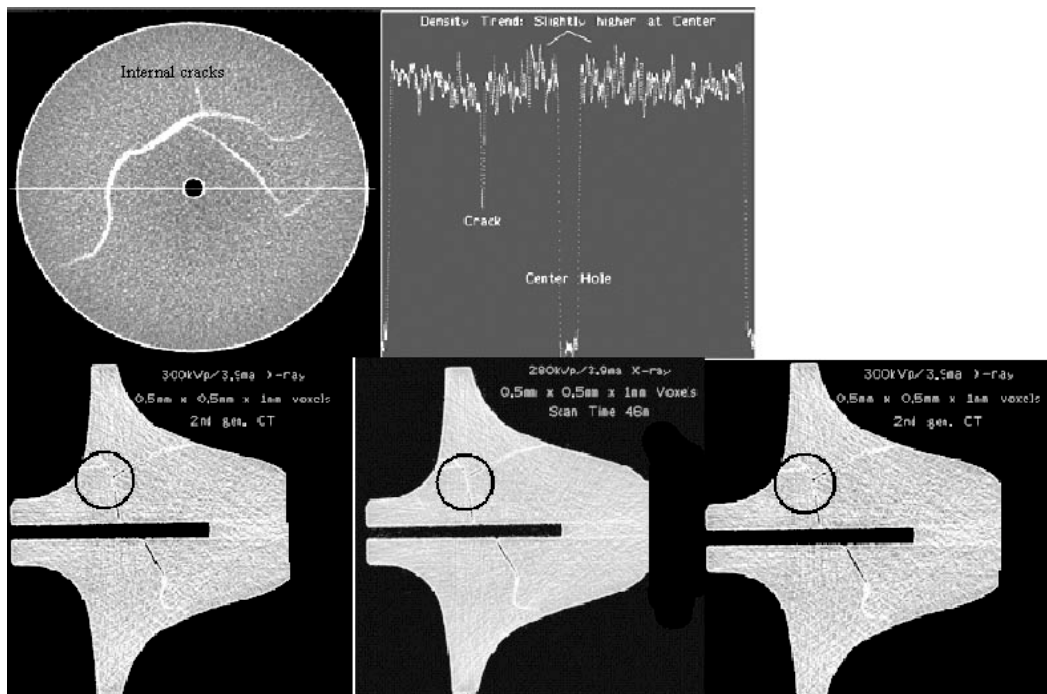
The evaluated ceramic materials include monolithic materials for turbine blades and nozzles and ceramic composite (CFCC) materials for combustor liners and transition ducts. The monolithic materials are Si_3N_4 , and the CFCC materials are both nonoxide (SiC/SiC) and oxide ($\text{Al}_2\text{O}_3/\text{Al}_2\text{O}_3$). The ANL NDE effort has included the development of (a) methods to determine thermal properties (thermal diffusivity) of the CFCC materials and conduct full-field mapping of thermal properties, (b) methods to detect delaminations of the CFCC materials, and (c) methods to detect surface cracks and subsurface cracks in the monolithic materials, as well as determination of whether the NDE data can be correlated with predicted component performance.

We have made significant progress in developing NDE methods for this project. Our most recent work has focused on composite ceramics. The thermal imaging method based on measurement of thermal diffusivity is now the NDE method being used to accept/reject components for use in the gas turbine. Full-sized liners (13 and 30 in. diameters and 8 in. length) have been successfully studied by ANL with the thermal imaging method. Tests have been conducted before service and after 2,000 hr of engine test time. The components are now scheduled for 4,000 hr of testing. By using a new, very-high-speed data acquisition system (up

3D X-Ray Tomographic Imaging System



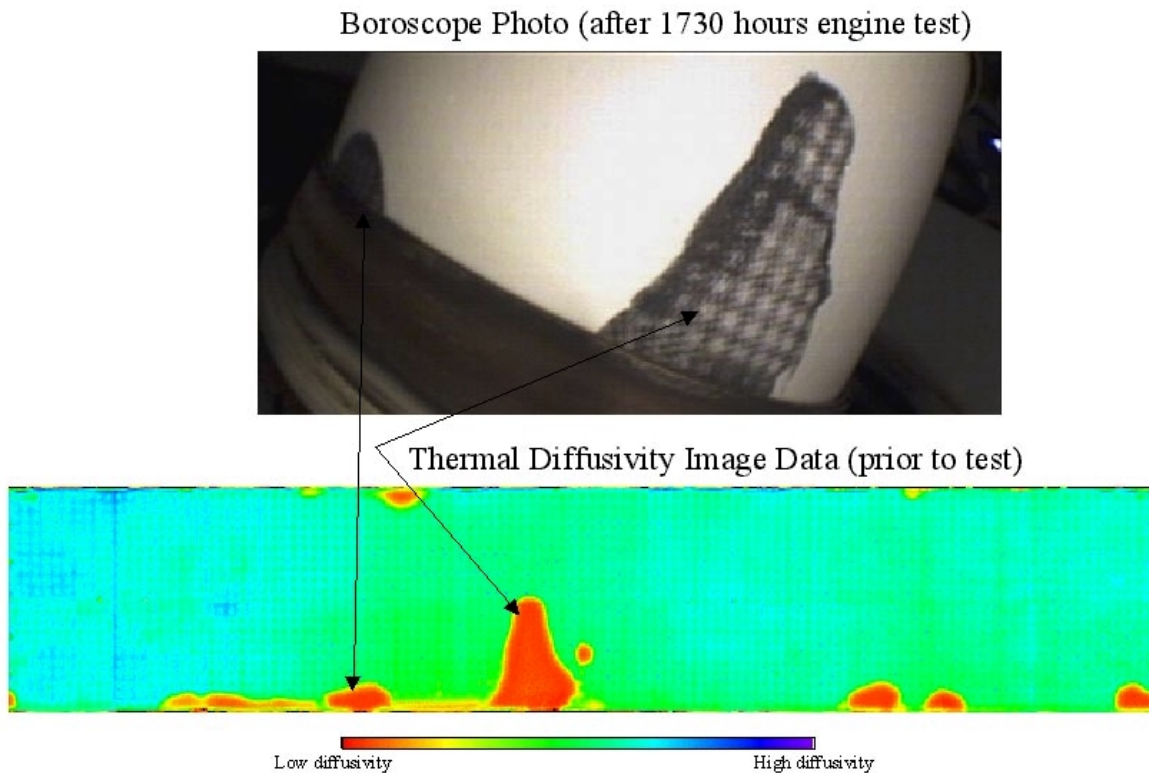
(a)



(b)

High-speed 3D X-ray tomographic imaging for characterizing ceramic components for microturbines: (a) schematic diagram; (b) X-ray CT image and detection of typical failure causing internal flaws ceramic turbine rotors

to 1900 frames/sec), including a new 12-bit dynamic range camera, we are developing one-sided data acquisition for complex-shaped components. Correlation of infrared imaging with new air-coupled ultrasonic technology has also been demonstrated, to allow confidence in the NDE data (see next figure). The air-coupled ultrasonic system operating at 400 kHz has a fast scan speed (>10 cm/sec), and we have added the capability to handle large cylinders of 13 and 30 in. diameters. Thus, the full-scale turbine engine components can now be examined. Results to date show excellent correlation to the thermal image data, and we can therefore replace the previous X-ray CT scan method for corroboration of data. X-ray CT data are still obtained to establish through-wall information.



Correlation of thermal imaging NDE data with field test data showing predictive capability

Development of NDE Technology to Detect Machining Damage in Monolithic Ceramics

The work in this project, funded by DOE/OATT/Heavy Vehicle Technology through Caterpillar, is a companion to the similar program described above and is also intended to develop NDE technology and methods for determining the extent of machining-induced damage in monolithic ceramic materials, primarily Si_3N_4 . In this work, the Caterpillar Technical Center is producing ceramic specimens under various machining conditions, including wheel speed, grit type and size, grit-to-wheel bond material, material removal rate, and downward force. The NDE method under development uses low-power lasers and special optical components in both the input and detector train. Data acquisition is totally automated, with the virtual instrument software package LabView serving as the user interface. The detector train further consists of two specially fabricated optical detectors to provide data from two numerical apertures. Polarization of the reflected light is also accounted for in the detector train. Output from the

two detectors is either summed or divided, and each resulting output is the used to form an “image” by a raster scan motion of the part under study. The resulting “image,” a speckle pattern of gray levels, is then manipulated by digital image processing. Specifically, the image is viewed as a two-dimensional array of values from which statistical data are derived. In this case, a standard deviation and a mean value are determined for each image. Dividing the standard deviation by the mean, we determine a coefficient of variation (COV) for each image, which represents some physical area on the specimen. Then, using the COV and plotting it as a function of machining conditions, we obtain extremely well-behaved correlation; Caterpillar is planning extensive further developments.

NDE Development for Thermal Barrier Coatings

This research effort is part of the OPT Advanced Turbine Systems (ATS) program to produce low-emission, high-efficiency gas turbines. We are developing NDE technology for application to thermal barrier coatings (TBCs). Usually of yttria-stabilized zirconia (YSZ), these coatings are applied to high-alloy, often single-crystal, substrates. The total coating system usually consists of the substrate, a bond coat, and the coating itself. These coating systems are being viewed in new turbine designs as the so-called prime reliant. That is, the coating system itself is mandatory for turbine system operation at design conditions. Because of the severe turbine engine operating conditions, which can include thermal excursions, foreign objects passing through the gas path and causing damage, and corrosion conditions, there is a need for NDE technology that can allow turbine system manufacturers and users to determine the condition of as-coated components, as well as to ascertain the condition of the coating during turbine operation. ANL is exploring two NDE technologies for this work. One is the use of elastic optical scattering, for which ANL has two patents wherein the optical translucency of the YSZ is used. That is, a low-power laser is incident on the coating and the light, after penetrating the YSZ, is reflected from the TBC bond coat interface. The scattering characteristics of the reflected light are characterized by polarizing filters and special detectors. The second NDE method is the use of flash thermal imaging, in which a fast (<10 msec) high-energy light is flashed on one side of the specimen and a fast (>1900 frame/sec) infrared thermal imaging camera captures the thermal decay signature. By using special software (locally written), we can determine changes in the thermal properties of the TBC and thus relate to debonds, changes in thermal properties, and so on.

Advanced Sensors for Real-Time Control of Advanced Natural-Gas Reciprocating Engine Combustion

This was a new project starting in FY 2000. The objective of this project is to develop advanced sensors for real-time combustion monitoring of advanced natural-gas reciprocating engines. The proposed development includes sensors to measure NO_x emissions and fuel composition. The sensors will then be integrated into an engine-combustion control system that can optimize engine performance and control NO_x emissions. To detect and monitor NO_x emissions in the range of 1-100 ppm, a nonradioactive ion-mobility spectrometer (IMS) will be developed. The IMS will detect negative NO_x ions in a high-pressure and temperature environment. A novel acoustic gas sensor based on speed-of-sound and acoustic relaxation spectroscopy measurement will be developed. The control system will use the IMS outputs to adjust the air/natural-gas ratio or fuel composition and thus optimize engine performance and control NO_x emissions.

This project will be carried out by a team of scientists and engineers from ANL, Northwestern University (NU), and Commercial Electronics (CE) of Broken Arrow, OK. ANL will conduct sensor development and testing, NU will carry out theoretical modeling for fuel-composition sensor development, and CE will provide design of control electronics and intelligent control systems.

DOE Office of Industrial Technologies

NDE Development for Continuous Fiber Ceramic Matrix Composites

The work in this effort is part of the DOE/OIT effort to develop materials for high-temperature applications as part of the Industries of the Future initiative. Work to date has focused on materials for the aluminum industry, for the Heat Treat initiative, and more recently for the pulp and paper industry

This work is directed toward development of NDE methods for three specific activities: (a) information for ceramic process development/reliability, (b) information to assess damage level from either oxidation or impact and (c) to establishing effectiveness of repairs. Work in this project is highly interactive with the industrial suppliers Honeywell Advanced Composites, Textron, McDermott Industries, B. F. Goodrich, and Composite Optics. Several special specimens were prepared by Honeywell to allow study of the thermal imaging and air-coupled ultrasonics relative to processing variables in the polymer impregnation process. Successful correlations have been established. Impact damage to SiC/SiC 2D laminated CFCC material, induced by instrumented pendulum impactors, is being studied by thermal imaging and air-coupled ultrasonic analysis for sensitivity to damage levels; initial correlations are excellent.

DOE Office of Science

Basic Sciences

NDE Technology for Joining

The research in this project is part of the DOE-SC/BES Center of Excellence for the Synthesis and Processing of Advanced Materials. Our work is directed toward development of NDE technology that can help characterize the joining of two materials. For example, in the joining of ceramic to ceramic, it is important to understand the effects of the wetting behavior on the complete infiltration of the material used for joining the two materials. Use of high-speed synchrotron X-ray radiation is being considered to study the time-dependent behavior of the infiltration. Once infiltrated, other much more macroscopic NDE technologies are being studied, including the understanding of the damping behavior through knowledge of the internal friction characteristics, high-frequency ultrasonic methods, and others.

Laboratory Technology and Research

Development of Rapid Prototyping Technology for Ceramics

This CRADA is a joint project with three small businesses; its purpose is twofold: (a) to develop technology that will allow the fabrication of net-shape or near-net-shape ceramics with

rapid prototyping (RP) technology, and (b) to develop technology to allow CAT-scanning image data to be used in reverse engineering for dimensional verification and quality verification of parts fabricated by rapid prototyping (RP).

Significant developments have been made in this project. First, in cooperation with Ford Advanced Manufacturing, an agreement was reached to allow testing of RP-produced ceramics in injection-molding facilities. Net-shape ceramic inserts were produced as part of a special die designed to allow testing of inserts. The complexity of the part shape and the finish of the surfaces required that green ceramic machining be developed for RP-produced ceramics. A precision three-axis milling machine was set up, software selected, and machining parameters established. Machining of both green as-cast and sintered ceramics was required. Two parts were sent to Mexico for full-scale injection mold testing. The ceramic inserts performed well enough to suggest that they should be certified for production, but this was not done. The parts were tested at full production rates for 25 min and no deterioration was observed. Second, work progressed on reverse engineering. The key to reverse engineering lies in the development of an algorithm that will allow data taken from a measurement system, e.g., a CAT scanner, a coordinate measuring machine (CMM), or laser rangefinder to be formatted for use as the input to solid-model CAD software programs. If automatable, this would provide the ability to replicate parts or obtain CAD designs from some measurement system. One such approach is constructive solid geometry (CSG). An approach was formulated at ANL using the CSG system for the algorithm. At present, the input to the algorithm is an algebraic boundary representation (as opposed to a parametric boundary representation). Algebraic boundary representations can be obtained from measurements with existing software. While not yet coded, the basic algorithm has been developed.

Development of Rapid Prototyping for Bioceramics

This CRADA project is a joint effort with two large businesses, Zimmer, Inc., of Warsaw, IN, the world's largest producer and supplier of orthopaedic devices, and Midwest Orthopaedics of Chicago, a leading orthopaedic surgical group. The purpose is twofold: (a) develop and evaluate RP technology for production of bioceramic prosthesis devices, e.g., small hand bones, and (b) determine the limitations of reverse engineering technology by using high-spatial-resolution CAT scanning. At present, two specific prosthetic devices, small hand bones and a selected forearm bone segment, have been identified for fabrication. Aluminum oxide has been formulated for rapid prototyping, and initial hand bones have been made. New materials of choice are hydroxyapatite/tricalcium phosphate because of its potential for surface-porosity control, which in turn should control engraftment time. Problems exist because of surface finish of currently produced materials, but may be overcome via five-axis machining or other surface modification methods. A high-density polymer skeleton model is being used to acquire 3-D X-ray CAT scan data. Using the hand and forearm bones, we have obtained CAT scans with very high (<60 μm) spatial features. These files have been subjected to digital image processing to allow computer files of the proper format to be electronically transferred to the RP machine, and initial bioceramic components have been produced. We have thus demonstrated almost all parts of the complete reverse engineering loop, leaving only reacquisition of the parts in order to compare dimensional fidelity.

DOE Office of Nuclear Energy, Science, and Technology: Nuclear Energy Plant Optimization (NEPO)

Steam Generator NDE Test Mockup Facility and Tube Degradation Data Base

The ability to assess the effectiveness of NDE techniques (both current and emerging) for detection and characterization of service-induced cracks in steam generator tubes is being addressed in this research program. The ANL steam generator mock-up with laboratory-grown stress corrosion cracks (SCC), and tubes with service-induced SCC from the McGuire nuclear power plant form the sample base for this effort. Maintaining interactions with industry is an important aspect of this program.

This current effort is focused on a review of emerging eddy current (EC) and ultrasonic NDE technology and the development of a library of well characterized, laboratory-generated axial, circumferential, inner-diameter (ID) and out diameter (OD) cracks for use in assessing advanced NDE methods being developed by DOE and EPRI. A facility in which tubes from service can be examined will be developed. Artifacts such as tube support plates, tube sheet, and deposits will be simulated in the facility. The primary focus of the work will be on the effectiveness of NDE techniques to characterize cracks.

Examples of in-service-cracked tubes that were obtained from McGuire 1 under an NRC program from the tube sheet roll transition will be examined. In-service cracks from the tube support plate region are also available. NDE data on the cracks will be obtained by a variety of NDE methods, including bobbin, EC array, and rotating EC probes operating over a wide range of frequencies; ultrasonic methods; and new candidate probes under development that are identified by DOE or EPRI as of interest. All of the raw NDE data, along with detailed calibration data, will be recorded on optical disks that can be made available to developers of new methods for analyzing NDE data. After the NDE examinations are completed, detailed destructive characterization of the specimens will be performed to identify and obtain depth profiles of all the cracks.

Advanced Eddy-Current Inspection System for Automatic Detection and Characterization of Defects in Steam Generator Tubes

The objective of this work is to develop software algorithms that will provide quick, accurate, and consistent detection and characterization of steam generator tubing degradation from data acquired from EC array probes.

A newly designed transmit-receive array probe has recently been introduced into the nuclear industry for steam generator tube inspection. This probe provides the unique capability of detecting tube degradation at all tube locations and operating at a high inspection speed. This probe has the potential of significantly shortening inspection schedules and reducing inspection costs, but its present limitation is that the data acquired can not be efficiently analyzed manually. Development of a method to automate the analysis of array-probe data will allow utilities to maximize the benefits of the probe, obtain consistent and repeatable results, and reduce the dependency and costs associated with large teams of data analysts.

Cooperation of the probe manufacturer in providing the necessary information on the probe design and data format is anticipated, given the dependence on automatic data analysis to maximize the benefits to the utility when using the array probe in the field.

DOE Office of Initiatives for Proliferation Prevention

Radioactive Incident Distance Monitoring (RIDIM) Sensor

This is an NIS-IPP Thrust I project conducted jointly with AOZT Finn-Trade of St. Petersburg, Russia, to investigate the technical and commercial potential of the NIS-developed RIDIM microwave radar sensor for radionuclide detection. The RIDIM radar is a result of 10 years of Russian research in the use of meteorological and air defense radar to detect low levels of airborne radionuclide emissions released from nuclear facilities. The technique involves illuminating a radioactive cloud with a microwave pulsed radar in the centimeter wavelengths; the radionuclides ionize the surrounding air and produce distinctive signal characteristics in polarization, scintillation, and reflectivity, which are exploited for detection, identification, and tracking. The uniqueness lies in the processing of the radar return signals. Finn-Trade mounted a 9.2-GHz radar on a van and remotely tested the detection capability of radioactive emissions during the operation of a nuclear power plant in St. Petersburg. Images of clouds over two power plant stacks, one idle and the other emitting, showed distinct contrasts for a emission level of 25 curies/24 hr. Further work is underway to lay the theoretical foundation for the refractive and scattering properties of radioactive clouds.

Work for Others

Department of Defense

Development of Fibrous Monolithic Ceramics/Solid Freeform Fabrication

This project is part of DARPA-funded work to develop a new higher-fracture-toughness ceramic material called fibrous monolith (FM), which has fracture characteristics similar to those for ceramic matrix composites, yet is made with conventional powders. In this cooperative effort with the Ceramics Section of ET, we have three goals: (a) develop technology to allow direct net-shape fabrication of components by using FM materials through solid freeform (SFF) fabrication methods; (b) develop technology to allow determination of properties, such as elastic modulus, for incorporation into analytical predictive models; and (c) act as principal contact with industrial collaborators to provide a well-defined end use component for the technology. The primary objectives of the overall project are to develop pressureless, sinterable oxide ceramics that can be fabricated by lower-cost manufacturing technology. In the SFF aspect, we have acquired a new high-pressure extruder head retrofitted SFF machine with special software for head control. The high-pressure extruder is needed because fabrication of the FM materials currently requires a central core of material surrounded by a thin outer shell. To fabricate a component made of FM material, a 1.5-cm-diameter billet about 5 cm long is produced with the central core and shell. This billet is then extruded through a high-pressure nozzle. We have successfully extruded and built components of ABS plastic materials that have been used to develop the tool path and nozzle control conditions. FM materials are just now becoming available for use in the machine. In the mechanical-properties aspect of the project, we are developing impact-excitation acoustic methods to determine elastic modulus. We have

assembled a well-defined impact pulse shape exciter and a noncontact capacitance microphone detector system, and we have recently incorporated a scanning laser vibrometer coupled to STAR Modal software package for mode shape analysis. Results have been obtained on several flat plates and the data compared to moduli obtained from 4-point flexure. We are currently comparing plate elastic modulus to individual flex bar modulus.

Millimeter- and Submillimeter-Wave Detection of Bioaerosols

This is a seed project from the U.S. Army Edgewood Chemical and Biological Center for evaluating the feasibility of millimeter- and submillimeter (mm and submm)-wave techniques for detecting bioaerosols associated with biological warfare (BW) agents. Proteins, DNA, and other biological molecules are known to exhibit rather large and distinct changes in dielectric properties in the mm- and submm-wave portion of the electromagnetic spectrum. Based on measurement of complex dielectric constants of these materials at different frequencies, our goal is to develop a portable cavity sensor for point detection. We will also test and identify unique spectral features that these molecules are theorized to exhibit due to resonance interactions of electromagnetic waves at these frequencies. Based on this strategy, our further goal is to develop a standoff detection sensor.

We tested the microwave and mm-wave absorption properties of two common biosimulants, *Bacillus subtilis* (known as Bg) and *Bacillus thurengiensis* (known as Bt). First we tested the dielectric properties of spore samples using a cylindrical cavity (TE_{01m}) in the 10-12 GHz range. The test samples supplied by the Army consisted of spore materials of various weights (3 to 9 mg) adhered to polycarbonate film. A convenient and reproducible way to expose the sample to the cavity field was to make a slit on one of its ends and test the effect of resulting evanescent waves on the film placed over the slit. The microwave absorption as measured by a change in quality factor generally increased with concentration; Bt was more absorptive than Bg at similar concentrations. Next, we tested the mm-wave properties of spores in a Fabry-Perot (F-P) cavity at frequencies of 254, 262, and 280 GHz. The film samples were not usable in the F-P cavity because the beam size was larger than the film area, causing the sample holder to interfere with the signal. Instead, we prepared our own samples from spores in freeze-dried powder form. The powder was adhered to a 0.1 in.-thick polypropylene (mm-wave transparent material) sheet over a 1-in.-diameter circular area by preparing a paste with water and then drying it overnight at 100°C. The sample regions consisted of 12 mg of Bg and 11 mg of Bt materials separated by a blank region. Millimeter-wave radiation from a tunable backward-wave oscillator source was coupled into the cavity with a beam splitter situated between the mirrors. A sample holder was set up inside the cavity for adjusting the sample plate to expose different regions. Fairly consistent and reproducible results were obtained; Bt once again generally showed more absorption than did Bg. Also, variations in absorption signal peaks were observed with respect to frequency. These results are preliminary and need further work in terms of calculating the exact dielectric parameters; however, the results show the potential of this technology for detecting biomolecules.

Nonintrusive Measurement of Temperature in Electroconsolidation® of Powder Preforms

This is a National Institute of Standards and Technology Advanced Technology Program (NIST ATP) project jointly with Superior Graphite Company, Northwestern University, and BioImaging Research Inc. to develop a nonintrusive method for intelligent process control of densification of powder preforms during Electroconsolidation. The three-year project was

successfully completed in November 2000. A key task in the project was developing a nonintrusive, real-time sensor to measure temperature in the Electroconsolidation die for monitoring and feedback control of sintering.

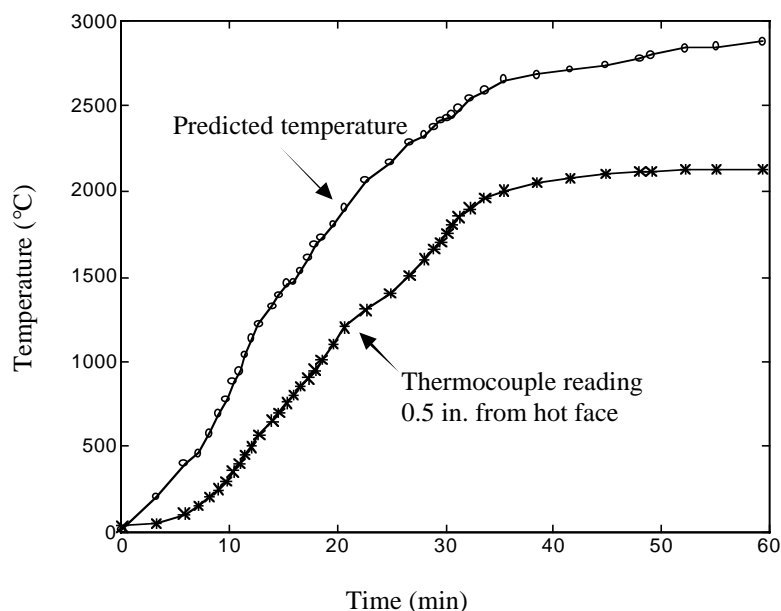
Electroconsolidation is a patented process developed by Superior Graphite for rapid pressure-assisted densification of preformed materials. The part to be densified is immersed in a granular graphite medium within a cylindrical die chamber, then pressed by hydraulic rams from the top and bottom of the die chamber while being resistively heated by electric current passing through the graphite medium. This is a simple and low-cost method of manufacturing complex-shaped parts from powders such as metals, ceramics, and polymeric materials. Cycle time is in minutes, compared to hours with the conventional methods such as hot isostatic pressing.

The main challenges to sensing temperature in the Electroconsolidation die are the extreme range of temperatures, the complex measurement environment, and the noninvasive, real-time measurement requirement. Temperatures in the die can reach 3000°C within a few minutes. The measurement environment consists of a graphite-particulate medium impregnated with multiple preforms and subjected to variable pressures up to 10,000 psi and currents up to 10,000 A. Because the local electrical properties of the graphite powder medium vary with pressure and temperature, wide temperature fluctuations can occur within the die chamber. Moreover, the geometry and location of the preforms, as well as their electrical properties, will affect the electric field distribution and hence the temperature profiles within the die. A real-time nonintrusive temperature sensor is needed for proper control of the temperature during sintering and to avoid over- or under-heating of the preform. No off-the-shelf sensors meet these EC measurement requirements.

We developed an ultrasonic pitch-catch temperature sensor in this project; it uses a pair of transmitting and receiving transducers at the top and bottom ends of the EC die, and a burst of ultrasound is transmitted across the graphite bed. The main design and implementation issues are the ability to transmit ultrasound through a graphite particulate medium in the presence of preforms, isolation of the transducers from the high process heat to keep it operating under the Curie temperature limit, measurement of transit time of the ultrasound pulses in the presence of extremely high currents and the resultant electromagnetic interference, and a means of correlating the velocity of sound with temperature under varying pressure and temperature that affects the elastic properties of the particulate medium. We developed a correlation between ultrasonic velocity in the graphite bed and its temperature by keeping pressure and heating rate constant or varying it in known steps. The sensor can predict average axial temperatures inside the die at up to 3000°C with an uncertainty of 2.5% (see next figure).

NASA

The objective of this work is to correlate data obtained from NDE methods with destructive analysis and with mechanical properties of ceramic matrix composite materials. In cooperation with NASA-Glenn Research Center and the NASA-Marshall Space Flight Center, SiC/SiC and C/SiC composites are being studied. In the work for NASA-Glenn, SiC/SiC plates of advanced ceramics are produced by NASA and studied at ANL using ANL-developed NDE methods. The plates are then sectioned for fatigue and creep tests at NASA. NDE data are acquired at different times during the fatigue and creep studies, Correlations are developed along with



*Ultrasonic prediction of temperature during sintering trial
of graphite puck in Electroconsolidation apparatus*

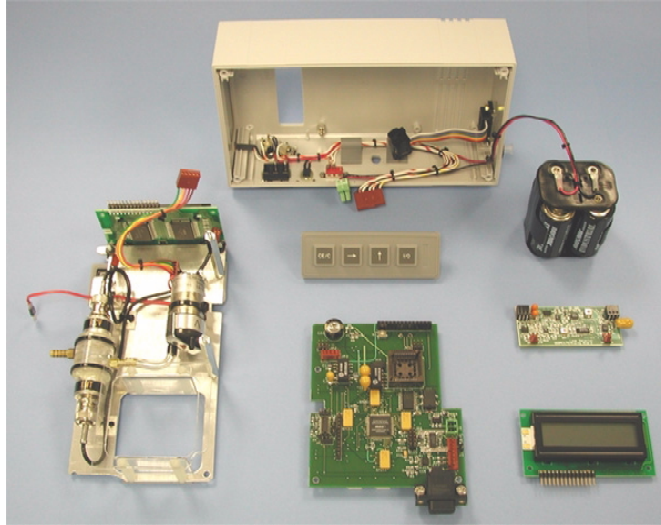
destructive sectioning on selected samples. The work with NASA-Marshall focused entirely on cyclic fatigue. ANL obtains NDE data and correlates the results with interrupted fatigue tests. Destructive analysis is also done on some samples at Southern Research Institute.

National Center For Manufacturing Sciences

Ultrasonic Helium Leak Detector

This is a work-for-others project funded by the National Center for Manufacturing Sciences (NCMS) of Ann Arbor, Michigan. The objective is to develop and demonstrate a portable speed-of-sound (SoS) helium leak detection instrument that can detect and locate leaks in jet fuel lines. In May 2000, ANL conducted a demonstration of the first prototype instrument for engineers at the Navy Airplane Depot (NADEP) in Jacksonville, Florida. That demonstration successfully showed that the instrument could detect and locate leaks with a 10^{-3} sccm leak rate. The instrument has a fast response/recovery time (<1 sec). The next two pictures show the prototype leak detector. The detector has the following features:

- DC power (two 9 V batteries or a rechargeable battery).
- One handheld box containing sensor, air pump, and control electronics.
- Microcontroller for signal analysis.
- LED indicator on safety glasses.
- Audio alarm with earphones.
- Zero button for reference calibration.



*Prototype leak detector disassembled (upper photo)
and in use (lower photo)*

Two prototypes were built; one was delivered to NADEP and was integrated with a smart charger developed by Vacuum Instrument to form a jet-fuel line leak detection system. The second prototype was delivered to Ford Motor Company. A patent application has been filed.

Nuclear Regulatory Commission

Steam Generator Tube Integrity Program

The Nuclear Regulatory Commission (NRC) program on the integrity of nuclear reactor steam generator tubing is continuing. Degradation of steam generator tubes is due to corrosion and wastage, pitting, denting, stress corrosion cracking, and intergranular attack. Cracking has

been experienced on both the primary and secondary sides of the steam generator tubes. Because knowledge about tube degradation in operating plants is derived from in-service inspections, the nondestructive test results and parameters from these inspections must be related directly to the failure pressures and leak rates or to the flaw characteristics that control these pressure and leak rates. The purpose of this program is to produce and/or update information, data, and predictive modeling for the degradation and inspection methods of current interest, i.e., various forms of cracks, multifrequency EC test equipment, modern probes and equipment, NDE parameters, and complementary inspection methods such as ultrasonic testing. Cracked tubing samples will be tested nondestructively before being subjected to pressure and leak rate testing.

Classical multifrequency and advanced EC tests will be conducted. This research will allow evaluations of various defect-specific management schemes and will support current NRC rulemaking needs and activities. Part of this effort involves the fabrication of a steam generator mock-up that will be used for round-robins and assessment of emerging technology to help establish the reliability of in-service inspection (ISI) and accuracy of sizing defects in steam generator tubing.

A primary objective of this multifaceted program is evaluating advanced NDE and signal analysis techniques for the reliable ISI of original and repaired SG tubes. Improved correlations between EC probe response and flaw morphology, leak rate, and failure pressure are being developed and validated. In addition, the reliability of the NDE parameters and techniques will be evaluated with respect to their ranges of applicability. The present research on improved ISI of SG tubes focuses on several primary areas. They include analytical/numerical electromagnetic (EM) modeling for the prediction of EC probe response, signal analysis techniques, flaw imaging and visualization methods, and evaluation of improved probe designs that use directional arrays so that defects of arbitrary orientation can be examined simultaneously.

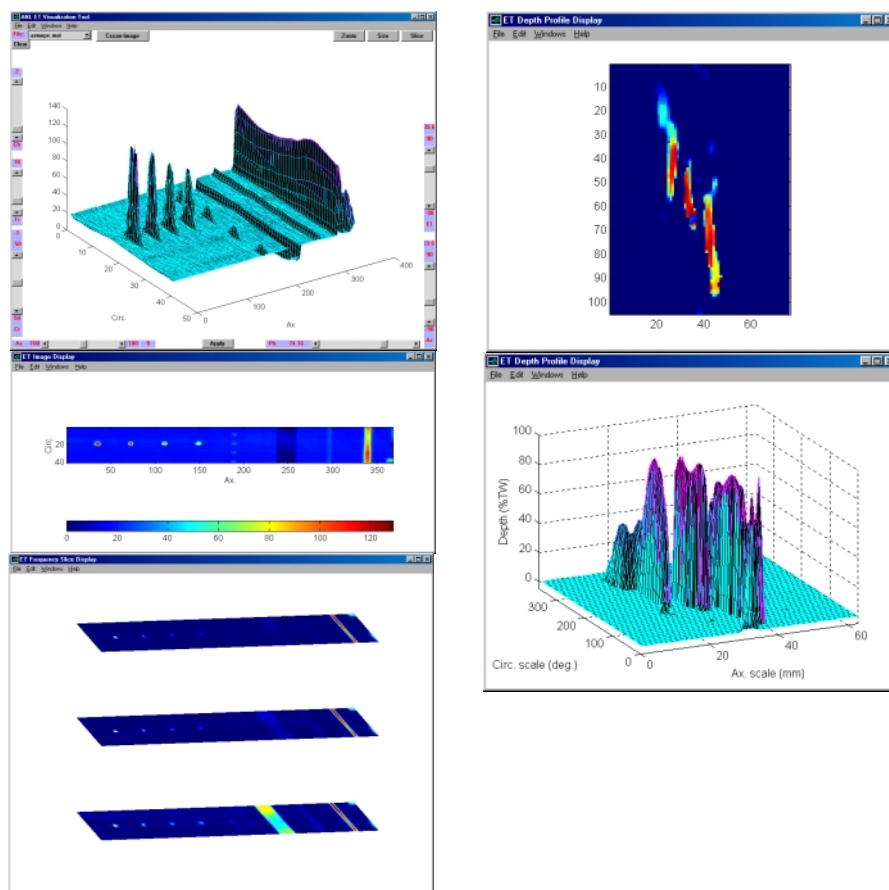
Studies are being carried out on the application of numerical electromagnetic EM models. To date, various analytical and numerical modeling approaches have been evaluated to the application of EC probe response for NDE of steam generator tubing. An elaborate 3D finite-element code is being used to study various EC inspection issues of interest. The results have contributed to a better understanding of EC probe response from complex flaw geometries through more accurate EM modeling. Such calculations can help in development of appropriate characterization schemes and can reduce the need for expensive experimental work. Furthermore, such models may also be used to develop a data base of simulated defects that can be used for assessing improved data analysis techniques.

Modern signal processing and computer-aided data analysis schemes are also being evaluated under this program. Multiparameter data analysis studies so far have dealt with implementation of multivariate linear (e.g., factor-based regression) and nonlinear (e.g., neural network) models to study potential correlations between EC signals and flaw size and tube structural integrity. Various scripts are being used to provide an automated means to efficiently format, calibrate, analyze, and visualize EC inspection results (see next figure). A series of algorithms have been implemented to carry out the conversion, normalization, and reformatting of EC readings for subsequent off-line analysis. An interactive tool is currently under development to incorporate all necessary data retrieval, calibration, and analysis routines in a

single virtual-instrument interface for more efficient data manipulation. This software currently allows processing of EC readings that are acquired with conventional inspection instruments.

Application of Neutron Diffraction to Determination of Residual Strains in Engineering Materials

Neutron diffraction techniques using ANL's Intense Pulsed Neutron Source and General Purpose Powder Diffractometer have been employed to measure residual strains in crystalline particles of a variety of engineering materials, with an accuracy sufficient to validate analytical models used to predict mechanical properties. The particles act as “strain gauges” for these materials. The stress-free temperature has also been measured by using neutron diffraction to determine bulk strain as a function of temperature (stress-free temperature is needed to predict mechanical properties, and the only practical way to obtain this experimental value is by neutron diffraction). In addition, the coefficient of thermal expansion as a function of crystallographic direction has been measured by neutron diffraction to characterize anisotropic effects. The most recent effort has involved the study of fabrication-induced strain in electrosleeved steam generator tubing.

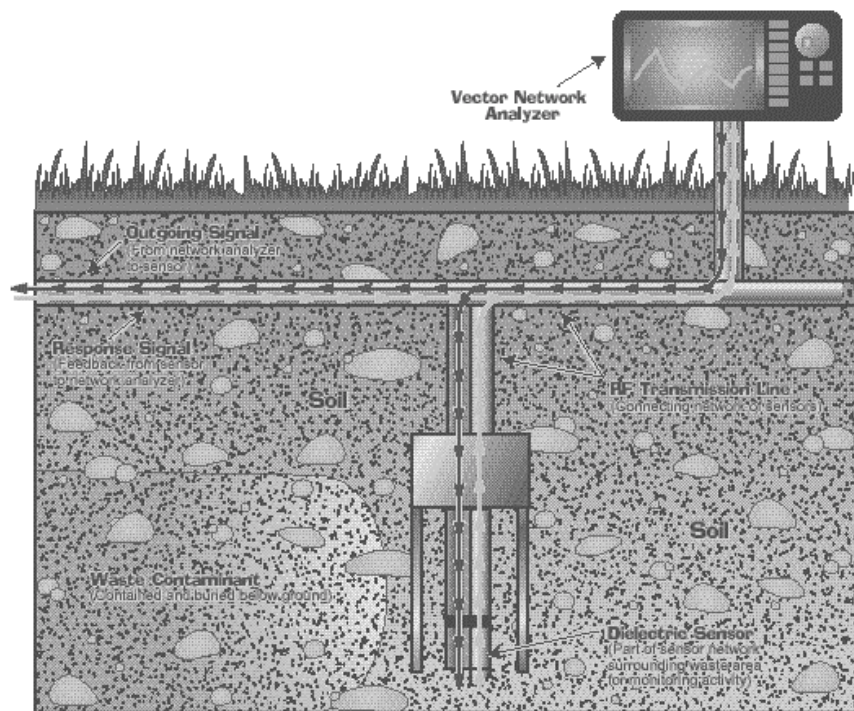


A series of MATLAB-based graphical user interface tools are currently under implementation at ANL to allow more effective visualization (left) and analysis (right) of NDE data from eddy current inspection of SG tubing

Laboratory Directed Research and Development

Radiofrequency Scan of Waste Containment Boundary

Many waste legacy sites, even after remediation, will require long-term monitoring (100 to 1000 years) to ensure that no leftover contaminants are released to local environments (groundwater and air). Engineered surface covers are being built to contain waste within remediated sites, and rugged, low-cost sensors are needed to monitor the integrity of the engineered covers over both the short and long term. This project investigates the concept of a network of radio frequency sensors, coated with a corrosion-resistant layer, for monitoring the migration of contaminant plumes. Input impedance of the network will vary with frequency and the dielectric properties of the soil medium, including that of contaminant plumes leached out of capped sites. The network is much like a telephone cable buried around the containment boundary, and the sensors are interrogated from one end of the cable (see next figure). The system is passive in that the line is not powered all of the time and provides measurements only on demand. As a result, it is a simple, inexpensive, and rugged network for monitoring the integrity of engineered covers over a long period. Preliminary tests, at 100, 500, 1000 MHz, of an open-ended coaxial probe buried in a sandbox showed good sensitivity to chemical contaminants.



Coaxial probe proposed for monitoring waste containment engineered surface covers over long periods of time

Cavity Ring-Down Sensor for Monitoring Oxidants in Cardiac Arrest Patients

Unexpected cardiac death, synonymous with ventricular fibrillation, is the single largest killer of both men and women in much of the world. More than half of the victims die within 1 hr of symptom onset. The chance of survival without external defibrillation is nearly zero. The

purpose of this project is to develop a biosensor based on cavity ring-down spectroscopy (CRDS) for measuring minute changes of free radicals that are generated at the cellular level under ischemic conditions such as cardiac arrest. Given the role that oxidants — by-products of free radicals — may play during ischemia/reperfusion, the development of human real-time oxidant biosensors to be used during the global ischemia of cardiac arrest could advance treatment in a significant way. Major oxidant markers of cell injury include hydrogen peroxide, pentane, and nitric oxide. Because of their high diffusivity, elevated levels (ppb range) of these oxidant markers are expected to be found in the exhaled air of cardiac arrest victims. Based on our preliminary work with millimeter-wave measurement of hydrogen peroxide, we will develop and test the feasibility and efficacy of a related ultrasensitive CRDS technique for real-time detection of key oxidant markers. The broad workscope, which will be carried out in collaboration with the Emergency Resuscitation Research Center of the University of Chicago, includes (a) design and construction of a prototype CRDS; (b) testing sensitivity, specificity, and response time of the technique using laboratory samples and cellular effluents; and (c) adaptation and testing of the sensor on human breath samples.

Myocardial Preconditioning Therapeutic Ultrasound

This is an exploratory project under a joint effort between ANL and The University of Chicago (UC). The purpose of this project is to examine the feasibility of using ultrasound to induce myocardial preconditioning. Myocardial preconditioning has been described as an adaptive response of the heart to brief episodes of ischemia that decreases necrosis during subsequent prolonged ischemia. Laboratory tests have shown that reactive oxygen species (ROS) such as superoxide, hydrogen peroxide, and hydroxyl radicals are generated from brief ischemia/reperfusion and that ROS are responsible for the initiation of preconditioning. Within the intact heart, possible sources of ROS include the cardiomyocytes, endothelial cells, neutrophils, or the auto-oxidation of catecholamines. Studies are needed to clarify the role of ROS as inducing agents and to identify their sources where they are metabolized. Use of hypoxia to induce preconditioning is the method being used in the laboratory. Testing has shown that 10 min of hypoxia in chick cardiomyocytes elicits a transient increase in ROS generation, predominantly hydrogen peroxide. However, hypoxic preconditioning may not be used as a clinical treatment due to the toxicity of hypoxia drugs and the difficulty of delivering the drug to specific ischemic myocardia due to low blood flow. Thus, there is a need to develop an alternative method of preconditioning protection.

The advantages of using ultrasound are that it is nontoxic and nondestructive. In FY 2000, we conducted feasibility tests at the UC medical school. Preliminary results show that ultrasound can induce release of hydrogen peroxide. The chick cells showed a similar response to that of hypoxia excitation. The response is directly proportional to the input ultrasonic energy and is slightly dependent on ultrasonic frequency. We also found that the response was instantaneous, compared to the 10 min delay with hypoxia excitation. We have therefore proposed a research project to (a) develop a high-power constant temperature ($\approx 36^\circ\text{C}$) focused transducer or transducer array and (b) conduct a parametric study of ultrasonic effects on myocardial preconditioning.

Multivariate Analysis for Decoding Chaotic ECG Traces of Sudden Cardiac Arrest

Although pacemakers and defibrillators have significantly improved the survival rate from cardiac arrhythmia, resuscitation by external defibrillators has shown little improvement over

the years and is still the topic of much discussion. In a collaborative effort with the Emergency Resuscitation Research Center of the Department of Emergency Medicine at the UC, feasibility studies are being carried out at ANL on the application of linear and nonlinear data analysis techniques to aid in interpretation of electrocardiogram (ECG) traces from cardiac arrest patients that could potentially improve the success of external defibrillation. Initial results of this ongoing effort have been quite encouraging in that they suggest presence of indicators in ECG signal that could point to differences between success and failure of defibrillation. The outcome of this research effort could help to both gain a better understanding of the complex dynamics of VF and aid in design and application of defibrillators for optimum resuscitation outcome.

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Development of Nondestructive Evaluation Methods for Hot Gas Filters

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H.-R. Lee and W. A. Ellingson

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Process for Controlling Flow Rate of Viscous Materials Including Use of a Nozzle with
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W. A. Ellingson and G. A. Forster

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No. 5,975,493 issued 11/2/99.

Transportation of Hazardous Materials

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The Transportation of Hazardous Materials (THM) Section provides direct technical support to DOE in reviewing Safety Analysis Reports for Packaging (SARPs) of radioactive material shipping packages for compliance with DOE Orders and with NRC and DOT regulations. In addition, the Section provides technical assistance to DOE on generic waste management and transportation issues, docket and data base management, guide and handbook development, training, and production and distribution of training and public information videos.

The Section provides direct technical support to NRC on developing improved guidance in the Standard Review Plan for License Renewal (SRP-LR) and the Generic Aging Lessons Learned (GALL) report for the review of license renewal applications for operating nuclear power plants. The Section also provides technical assistance to NRC on the review of license renewal application for the Arkansas Unit-1 (ANO-1) nuclear power plant, the review of current licensing actions, and other regulatory improvement activities.

Review of Aging Issues for License Renewal

A substantial number of the 103 operating nuclear power plants in the United States will reach the end of their initial 40-year licensing periods in the next 10-15 years (see graph on next page), and the NRC is developing criteria for license renewal. NRC is sponsoring a program at ANL for the review of generic aging issues and the evaluation of existing aging management programs for the various aging effects on the systems, structures, and components in nuclear power plants. NRC is particularly interested in developing improved guidance that enhances the efficiency and effectiveness of the staff review of license renewal applications. NRC has actively engaged the stakeholders and the public in the development of the improved guidance documents for license renewal.

Over the years, ET and the THM Section staff (O. K. Chopra and D. C. Ma) have worked with NRC on projects that exhaustively documented the industry and the NRC positions for the various aging effects and mechanisms for all passive, long-lived components subject to aging degradation in light water reactor (LWR) operating environments. Many of these

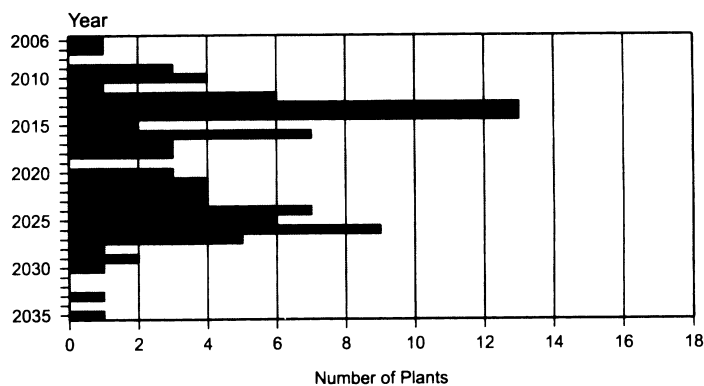
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*U.S. commercial nuclear power plants reaching
40-yr design lives (2006-2035)*

positions are based, in part, on the NRC-sponsored research at ET in areas such as thermal aging embrittlement of cast austenitic stainless steel, environmental assisted cracking, and steam generator tube integrity, etc. Two reports documenting the earlier work were published in 1996, "Summary of Technical Information and Agreements from Nuclear Management and Resources Council Industry Reports Addressing License Renewal," NUREG-1557, and "Nuclear Power Plant Generic Aging Lessons Learned (GALL)," NUREG/CR-6490. A draft Standard Review Plan for License Renewal was prepared by Section staff and published in 1997. Section staff have also reviewed numerous technical reports submitted by the PWR and BWR Owner's Groups and utilities that addressed aging degradation of major reactor components and systems and the impact on license renewal. For example, two years before Baltimore Gas and Electric (BG&E) submitted its license renewal application to NRC for the Calvert Cliffs Units 1 and 2 (845 MWe each), Section staff prereviewed over 20 BG&E "Aging Management Review Reports" and generated hundreds of Request for Additional Information (RAIs) for NRC. Calvert Cliffs is the first U.S. nuclear power plant that received a license extension (in May 2000), and ANL's contribution was acknowledged in the NRC's Safety Evaluation Report for Calvert Cliffs, NUREG-1705, December 1999.

While the NRC staff began reviewing the first two license renewal applications for Calvert Cliffs and Oconee plants in May 1998, Section staff have begun the development of a "Generic Aging Lessons Learned (GALL)" report, NUREG-1801, and the Standard Review Plan for Review of License Renewal Applications for Nuclear Power Plants (SRP-LR), NUREG-1800. Although the titles of these reports remain the same as those of the earlier reports, their purposes and contents are very different. The principal purposes of the SRP-LR are to ensure the quality and uniformity of staff reviews and to present a well-defined base from which to evaluate the applicant programs and activities for the period of extended operation. The SRP-LR is also intended to make information about regulatory matters widely available, to enhance communication with interested members of the public and the nuclear power industry, and to improve their understanding of the NRC staff review process. The SRP-LR has been developed to focus the guidance for NRC staff review on areas where existing aging management programs should be augmented for license renewal. The technical basis in the SRP-LR for the review of aging management programs is documented in the Generic Aging Lessons Learned (GALL) report, NUREG-1801. The GALL report would be referenced in the SRP-LR as a basis for determining the adequacy of existing aging management programs.

The adequacy of the existing aging management programs for the systems, structures and components are evaluated rigorously against each of the 10 elements (or attributes) delineated in the SRP-LR: (1) Scope of Program, (2) Preventive Actions, (3) Parameters Monitored/Inspected, (4) Detection of Aging Effects, (5) Monitoring and Trending, (6) Acceptance Criteria, (7) Corrective Actions, (8) Confirmation Process, (9) Administrative Controls, and (10) Operating Experience. Altogether, 45 existing aging management programs were generically evaluated for mechanical, structural and electrical components. These generic aging management programs include, for example, ASME Section XI inservice inspection, reactor water chemistry, BWR stress corrosion cracking, PWR vessel internals, steam generator tube integrity, flow-accelerated corrosion, bolting integrity, fire protection, structures monitoring, electrical cables and connections.

The evaluation results documented in the GALL report indicate that many of the generic existing programs are adequate to manage the aging effects for particular structures or components for license renewal without change. The GALL report also contains recommendations on specific areas for which generic existing programs should be augmented for license renewal. An applicant may reference the GALL report in a license renewal application to demonstrate that the programs at the applicant's facility correspond to those reviewed and approved in the GALL report and that no further staff review is required. If the material presented in the GALL report is applicable to the applicant's facility, the NRC staff should find the applicant's reference to the GALL report acceptable. In making this determination, the NRC staff considers whether the applicant has identified specific programs described and evaluated in the GALL report. The NRC staff also ensures that the applicant verifies that the approvals set forth in the GALL report for generic programs apply to the applicant's programs. The focus of the staff review is thus on the augmented aging management programs for license renewal. Because of this focus, the incorporation of the GALL report information into the SRP-LR should substantially improve the efficiency and effectiveness of the license renewal process.

NRC has been actively engaging stakeholders and the public from the beginning of the development of the improved guidance documents in the GALL report and the SRP-LR. Numerous public meetings and workshops were held in the last three years to solicit stakeholder inputs, and Section staff have participated in all of the major public meetings and have prepared the majority of the preliminary recommended NRC staff dispositions of public comments contained in the five appendices of "Analysis of Public Comments on the Improved License Renewal Guidance Documents," NUREG-1739: Appendix A addresses the participant comments from the license renewal public workshop on September 25, 2000; Appendix B addresses the written comments submitted by the Nuclear Energy Institute (NEI); Appendix C addresses the written comments submitted by various stakeholders such as utilities, Nuclear Information and Resource Service, and private citizens; Appendix D addresses the comments submitted by the Union of Concerned Scientists (UCS); and Appendix E addresses the Advisory Committee on Reactor Safeguards (ACRS) consultant comments on the structural and electrical chapters in the GALL report. More than 800 stakeholder comments were addressed in NUREG-1739.

The March 1, 2001, drafts of the GALL report and the SRP-LR have been submitted to the ACRS with meetings scheduled in April 2001. As the experience of utilizing GALL and SRP-LR accumulates in reviewing plant license renewal applications in the foreseeable

future, Section staff are likely to be asked by NRC to update these guidance documents periodically to capture the lessons learned and improve the guidance documents for license renewal.

Extension of the operating license for 20 years for Calvert Cliffs Units 1 and 2 means ≈ 28.7 GWe (assuming 85% plant capacity factor) electricity that does not have to be generated from other nonnuclear sources and thus the elimination of the associated carbon burden to the environment. Section staff have contributed directly to that achievement. Keeping the 103 nuclear power plants running in the U.S. for an additional 20 years beyond their original 40-year design lives can have a tremendous socioeconomic impact and environmental benefit to the country.

SARP Review and Technical Assistance

The SARP Review Group provides DOE with an independent review and evaluation of the information contained in a SARP that describes the packaging used to transport radioactive materials. The review is conducted to apprise DOE of the ability of the packaging to meet all DOE orders and federal regulations for safe transport. The process ensures that a consistent and independent review is performed, while maintaining the authority granted to DOE by the U.S. Department of Transportation for the packaging evaluation and certification.

The SARP Review Group operates independently of other Section programs and comprises a staff encompassing the disciplines and experience necessary to perform in-depth review and confirmatory evaluation of the entire range of information provided in a SARP, which is a voluminous document consisting of nine chapters: General Information and Drawings, Structural, Thermal, Containment, Shielding, Criticality, Operating Procedures, Acceptance Tests and Maintenance Program, and Quality Assurance. Together these chapters in the SARP should provide technical evidence demonstrating that the package meets all applicable safety standards and regulations. The primary goal of the review is to verify and ensure that the packaging will perform as intended under both the normal conditions expected in transport and the hypothetical accident conditions. These transport conditions are detailed in the regulations contained in Title 10 of the Code of Federal Regulations, Part 71 (10 CFR 71). The regulations contain specific environmental and mechanical loading conditions that the package must be able to accommodate during transport. Because of the hazardous nature of the package contents, the regulations are necessarily severe. Consequently, each element of the review is essential and requires, more than other programs, an integrated team effort from the SARP Review Group.

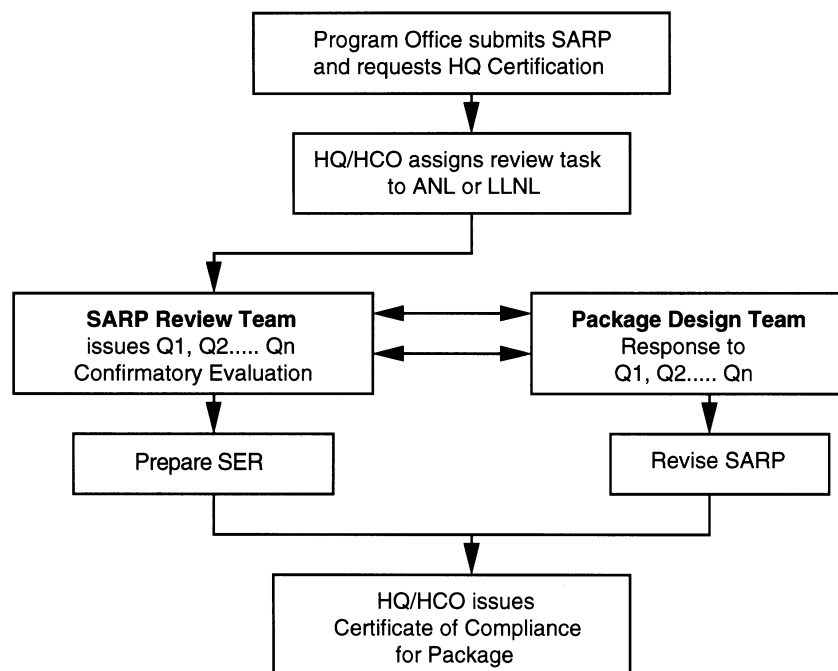
Last year the Group reviewed five SARPs for various radioactive material contents including low-enrichment uranium ingots, source capsules, highly enriched uranium metals and oxides, plutonium metals and oxides, and actinides. The packages of these SARPs are sponsored by five DOE Offices (EM, DP, MD, NE, and NN) and their certifications invariably lie on the critical paths of major DOE programs in environmental remediation, site cleanup and closure, and fissile material disposition. On the low-enrichment uranium ingots, for example, the Section's work in the last two years enabled more than 400 shipments of 3,277 MTU transferred to Portsmouth, Kentucky. Of the more than 20 different payload types required by the Fernald site in Ohio, 17 have been received as a direct result of the technical

review support provided by the Section. This work has earned DOE recognition and kept the project on schedule to complete the disposition of the nuclear product material at the Fernald and Hanford sites.

Technical Assistance Activities

As mentioned earlier, the THM Section staff in the SARP Review Group provide technical assistance to DOE in several areas, including guide and handbook development, training, and other technical support as required by DOE. Section staff have been involved in the review of (a) the NRC Standard Review Plan for Transportation for Radioactive Materials (NUREG-1609), (b) the DOE Criteria for Safe Storage of Plutonium Metals and Oxides (DOE/STD-3013), (c) the DOE Order 460.1B on Packaging and Transportation Safety, (d) the DOE Packaging Review Guide, (e) the IAEA Regulations for the Safe Transport of Radioactive Materials, ST-1, and (f) various Federal Notices of Rule Making activities on transportation of radioactive materials by DOT and NRC. Section staff have also been involved in the preparation of the DOE Radioactive Materials Packaging Handbook (ORNL/M-5003), and Reengineering EM's Package Certification Program, DOE/EM-0383, which was published in November 1998.

The goal of reengineering DOE's Package Certification Program is to enhance process efficiency in order to meet an expected increase in workload in future package certification. Because SARP review is the major activity supporting package certification, Section staff were deeply involved in the reengineering of the certification process, which is shown in a simplified flow chart in the next figure. The process begins when a DOE program office submits a SARP and requests DOE-HQ certification of the package. Depending on workload, the HQ Certifying Official (HCO) assigns the SARP review task to either ANL or LLNL. Once the technical review of a SARP starts, it becomes an iterative process of questions and



Simplified flow chart for the DOE package certification process

responses between the SARP review and package design teams until all issues are resolved, and the reviewers have verified, through independent confirmatory evaluation, that the SARP has indeed demonstrated package compliance to all applicable safety regulations. The outcome of the SARP review is documented in a Safety Evaluation Report (SER) that provides the basis for issuing a Certificate of Compliance for the package. The SER is part of the approval record for the package and is accessible to the public (it is posted at www.rampac.com).

Despite the best efforts by the SARP review teams to streamline the review, package certification ultimately depends on the design team that must prepare and/or revise a SARP that demonstrates package compliance to all applicable safety regulations. During the reengineering workshop in August 1998, the participants were asked for their ideas for improvement of the package certification process. More than 40 ideas were generated and ranked according to the three criteria: (a) relative ease of implementation, (b) crucial or most important, and (c) most cost-effective. It is of interest to note that "Provide training for package designers and new SARP applicants" came out on top of the crucial or most important idea for process improvement, whereas "Update the package review guide more often or send out new issues list more frequently" was a close second. The consensus of the workshop participants thus echoes one of the main conclusions in the ANL white paper that states "Training courses and guidance documents are the most effective way in improving the quality of SARPs by training the people and providing them with reference materials in SARP preparation and review. DOE/EM-5 needs to reach out to members of the DOE packaging community by providing regular training and guidance, because the efficiency of the packaging certification process cannot be improved unless the quality of initially submitted SARPs is substantially improved." The importance and the need for providing guidance documents and training in SARP preparation and review was a recurring theme at the DOE Packaging Management Workshop, which the Section hosted at ANL on October 31-November 1, 2000. The workshop was attended by more than 50 Headquarters, Field, and Contractor program managers across the DOE complex.

Training Courses

Since the early 1990s, Section staff in the SARP Review Group have developed three training courses for DOE: (a) Application of the ASME Code to Radioactive Material Transportation Packaging, (b) Quality Assurance to Radioactive Material Packaging, and (c) Hydrogen Gas Generation in Radioactive Material Packaging. Section staff developed these courses based on the review experience of more than 35 SARPs, which show that the majority of the review questions are in the structural chapter ($\approx 27\%$ of 2,288 questions total between 1987-94); general information, containment, operating procedures, and quality assurance chapters each received $\approx 10\%$ or more of the questions. The statistics of the Section's SARP review thus clearly indicate where training should be conducted.

The ASME Code Course

Section staff presented the first ASME Code course in March 1994 in Germantown, MD, to a select group of DOE and NRC personnel. The purpose of that course was to gauge its effectiveness and obtain feedback from knowledgeable regulators. Since then, the course has been thoroughly updated to the latest Codes and Federal Regulations. As presently constituted, the 2-day ASME Code course consists of 20 lectures taught by four Section staff

(D. T. Raske, R. R. Fabian, D. R. Henley, and R. Seidensticker), a LLNL SARP review staff, and one outside expert who is either from NRC or an independent consultant.

Both NRC and DOE recommend Section III of the ASME Code for radioactive materials transportation packagings because it is the only available authoritative guidance for the design, fabrication, examination, and testing of nuclear power plant components. However, designers and fabricators of transportation packagings are usually not too familiar with similar activities covered in the ASME Codes for nuclear power plants. Thus, the overall objective of the course is to show the participants how to design, fabricate, examine, and test a transportation packaging so that it meets all governing federal regulations and applicable ASME Code requirements. The course includes lectures on specific topics such as materials of construction, design and stress analysis, bolting design and analysis, brittle fracture considerations, fabrication, examination, and testing.

Design and construction of a transportation packaging to the requirements of the ASME Code is only one step in a process that results in the certification of a package for shipping radioactive materials. Demonstration of package compliance to all applicable safety regulations can be done by analysis (using the ASME Code procedures) or by physical testing. Even though qualification by testing usually does not require detailed stress analysis of the structural components of the package, the materials of construction must be rigorously pedigreed following the ASME Code or equivalent materials specifications. Thus, qualification by testing must be supported by a comprehensive quality assurance plan to provide materials traceability, documentation of construction methods, and documented evaluation of test results. The ASME Code course discusses physical testing as the other acceptable method of qualification and demonstrates how the applicable ASME Code rules are applied for these cases.

The Quality Assurance Course

Since 1987, Section staff (R. R. Fabian, D. T. Raske, J. J. Oras, R. Seidensticker and L. Garrison) have presented the QA training course 29 times to more than 650 participants. (The most recent session was at ANL in November 2000.) Over the years, the course has evolved with time and changes in Codes and Regulations such as DOE Order 460.1A and the ASME/NQA-1. However, the course has always emphasized the development of a QA plan for a package and a QA chapter in a SARP based on a graded approach according to the importance to safety. The main objective of the course is to teach techniques for the development of a QA plan at the onset of a project, which aids in the establishment of effective QA requirements in the QA chapter on all aspects of a packaging design, fabrication, testing, maintenance, repair, operation, document control, verification, etc. Other issues discussed in the course also include compliance-based versus performance-based QA programs, grandfathering, and the role of QA in the life cycle of radioactive material packagings.

The ASME Code and the QA courses supplement each other in many ways. Meeting the ASME Code requirements depends on having a thorough QA program for the package. As an example, when an older packaging was designed and constructed under a robust QA program, the materials used in the packaging construction, even though they may not be specified by the ASME Code, should have a pedigree that is often sufficient to satisfy Code rules to be acceptable and provide the required safety margins.

Hydrogen Gas Generation Course

This course has been offered five times since the early 1990s and is now undergoing major revision before it is presented again. Revision is necessary because of the complexity of the issues that escalated in recent years due to the concerns for long-term storage of plutonium-bearing materials, as well as the need to transport such materials after long-term storage for treatment, use, or disposal.

For several years, Section staff have addressed the issues related to hydrogen gas generation in transportation packagings containing plutonium metals and oxides. The phenomenon involves alpha radiolysis of moisture in the plutonium compounds and/or any other adjacent hydrogenous materials, chemical reactions of plutonium compounds and other packaging materials with water and with oxygen, gas phase reactions and recombination, and potential for hydrogen combustion, deflagration and detonation in the packaging. Section staff (J. J. Oras, A. B. Rothman, and Y. Y. Liu) have been closely involved in all aspects of the hydrogen gas generation issues and recently collaborated with the LLNL SARP Review Group and developed a draft DOE Regulatory Review Guide for Gas Generation Issues related to Transportation of Plutonium Metals and Plutonium Oxides. The Section also hosted a DOE/EM-5 Licensing Meeting at ANL in February 2001 to discuss the gas generation issues for the review and certification of two transportation packagings (9975 and SAFKEG). The certification of these two packagings lie on the critical path for the disposition of the plutonium-bearing materials currently located at Rocky Flats, Colorado; these materials are to be packaged and transported to the Savannah River Site starting in 2002.

Criticality Safety

The objective of the criticality safety evaluation of a SARP is to verify that the packaging design satisfies all of the criticality requirements and that the package will perform as intended under normal conditions of transport and hypothetical accident conditions defined in 10 CFR 71. Under normal conditions of transport, a package used for shipment of fissile material must be so designed and constructed and its contents so limited that it would be subcritical if water were to leak into the containment system or if its liquid contents were to leak out. In addition, the maximum reactivity of the fissile materials is to be attained with the most reactive credible configuration, with moderation by water to the most reactive credible extent, and with close reflection by water on all sides. Under hypothetical accident conditions, the package must remain subcritical with the fissile material in the most reactive credible configuration consistent with the damaged condition, with water moderation to the most reactive credible extent, and with reflection by water on all sides as close as is consistent with the damaged condition.

Among the SARPs reviewed by the Section last year, the Steel-Banded Wooden Shipping Containers (SBWSC) are used for shipment of more than 30 types of unirradiated, low-enrichment (up to 1.25 wt.% U²³⁵) uranium metal ingots. Because the wooden containers will burn in a hypothetical fire accident, the safety review of the package essentially amounts to a criticality safety evaluation of arrays of uranium ingots that are "scattered and arranged" in the most reactive configuration, as required by 10 CFR 71.55 and 10 CFR 71.59. Section staff have performed many Monte Carlo calculations for infinite and finite arrays of ingots using the MCNP (continuous energy cross sections) and the KENO (multi-group cross sections) codes. From the massive set of calculations, Section staff (Liaw, Turski, and Liu) have also

developed a systematic framework for the treatment of classes of ingots that significantly reduces the burden of detailed analysis. Dr. Liaw presented this work at the Annual Meeting of the American Nuclear Society (ANS) in San Diego, CA, June 2000, which became part of a feature article in the August 2000 Issue of Nuclear News.

Tokaimura Criticality Accident Analysis

On Thursday, September 30, 1999 at about 10:35 a.m. Japan time, a criticality accident occurred in a room at the fuel conversion plant in Tokaimura, Japan. Workers at the plant were pouring buckets of uranyl solution into a large steel precipitation tank. Each bucket contained ≈ 2.4 kg of enriched uranium (18.8% U-235). The workers did not follow the plant criticality safety procedure on feeding the uranyl solution into the precipitation tank. A chain reaction occurred when the seventh bucket was poured and the workers saw a blue flash emanating from the tank. According to news reports, all three workers in the vicinity of the tank received high radiation doses, i.e., at least 800 rem for two workers who went into shock and had diarrhea, fever, high white-blood-cell count, and reddened skin — all symptoms of acute radiation sickness. The radiation level on the plant grounds was 10,000 to 20,000 times normal as of 5:00 p.m. Thursday. Thirty-six other workers on-site received abnormal doses, as did three paramedics and seven residents near the plant. Approximately five hours after the accident, the 160 residents who live close to the plant (within 300 m) were told to evacuate to a community center. Later that day, 300,000 residents in the neighboring towns within a 10-km radius of the plant were warned to stay inside closed doors and sealed windows. Criticality reaction in the tank continued on and off over a 17-hour period until the water in the jacket surrounding the tank was drained.

Based on the information gathered from the news reports after the accident, Section staff (Jay Liaw) performed a quick assessment of the accident that provided technical insight on the criticality accident and its radiological impact to the workers and the environment. The results can be summarized as follows:

(a) Energy Release: Based on similar criticality accidents in fuel processing facilities in the past and an assumption of 10^{17} fissions during the initial burst, the estimated total energy release was ≈ 3.2 MW-sec, or a power burst of 3,200 MW in ≈ 1 msec. If this energy were imparted adiabatically to the uranyl solution, the temperature rise would be $\approx 20^\circ\text{C}$. This power burst may have caused some evaporation of the solution and bubbles may have been released from the evaporation and the gaseous fission products in the tank. Subsequent cooling and condensation could repeat the criticality reaction in the tank until the water in the jacket was drained 17 hours after the initial burst.

(b) Prompt Neutron and Gamma Radiation Doses: Based on the geometry and construction materials of the tank and standard values for the physics parameters (e.g., fission yields, removal cross sections, attenuation coefficients, buildup factors, and flux-to-dose conversion factors), the radiation doses to the workers from prompt neutrons and prompt gammas were calculated for an initial burst of 10^{17} fissions. The results are ≈ 300 and 450 rem for prompt neutron and prompt gamma radiation doses, respectively, giving a total prompt radiation dose of 750 rem to the workers in the vicinity of the tank. This estimated value compares reasonably well with the reported value of 800 rem received by the two workers who were critically injured during the initial burst of the accident and later died.

(c) Fission Product Inventory and Delayed Radiation Doses: The ORIGEN code was used to calculate the fission-product inventory from the fissioning of 10^{17} atoms of U-235. At the end of the initial burst, the total calculated fission product radioactivity was $\approx 3.8 \times 10^5$ curies, which decays rapidly (by a factor of 10,000) to ≈ 40 curies after 2 hr, ≈ 4 curies after 1 day, and ≈ 0.5 curie after 5 days. Gaseous and volatile fission products such as Br, Kr, Sr, I, Xe, and Cs constituted a very significant portion of the initial radiation release shortly after the burst. The delayed gamma doses (from the fission products) received by the rescue paramedics rushed to the scene during the first 10 min after the initial burst is estimated to be ≈ 20 rem. The calculated decay characteristics are in agreement with the reported time variation of the radiation levels at the plant. Residents near the plant should not have received any prompt doses from the criticality accident.

The major conclusions from the quick assessment, completed two weeks after the accident, were all confirmed by the international expert panel sent to investigate the accident six months later. Dr. Liaw was invited by the Chicago Section of the ANS in November 1999 as a featured speaker on the subject; his talk was well received by the participants that evening.

Other Activities

Section staff have been involved in other activities for the development of new programs based on the Section's strength and experience. The activities are (a) conversion and use of depleted uranium hexafluorides (A. B. Rothman and Y. Y. Liu), (b) biodegradation of oil spills in the ocean (A. B. Rothman and Y. Y. Liu), (c) impregnation of porous silicon for nanocomposite materials (S. W. Tam), and (d) development of the U-Mo-Ti alloy fuel ring for the pulse reactor SPR-III (V. N. Shah). Section staff have developed proposals for the conversion of DUF_6 into mineral forms for disposal, or into depleted uranium metals and oxides for beneficial use as shielding material in spent-fuel casks and by-products such as HF and CaF_2 . One proposal has been funded by the DOE Initiative for Proliferation Prevention (IPP) Program; two invention reports are pending for the beneficial use of depleted uranium as shielding material and for biodegradation of oil spills in the ocean.

Recent Work by the Transportation of Hazardous Materials Section

Publications

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J. R. Liaw and Y. Y. Liu

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QA Chapter Development

R. R. Fabian

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Developing a Project Quality Assurance Plan

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10 CFR Part 71, Subpart H vs. DOE Order 414.1

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ASME NQA-1, Element 3 Design Control

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Typical Packaging QA Problems

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Training Course Overview

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DOE Order/Regulations

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Compliance vs Regulations

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Developing of QA Plan

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Materials Technology

Ceramics

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The Ceramics Section investigates the synthesis, fabrication, and characterization of ceramic materials to optimize their properties for practical engineering applications. In most cases, the applications are intended to facilitate the generation, transmission, storage, distribution, and conservation of energy. For example, the goal of the superconductor program is to develop ceramic superconductors for more efficient generation, transmission, storage, and distribution of electrical energy. Ceramic membranes are being developed to separate hydrogen from product gases in a coal gasification plant. The cementitious properties of phosphate-based materials are used to stabilize radioactive and chemical wastes. These phosphate-based materials have found other applications, for example, in the waste recycling and biomedical fields. In the structural ceramics/composites program, more reliable and less-expensive structural ceramics, composites, and thermal-barrier coatings are being developed for use in advanced high-efficiency gas turbines and other applications. Hybrid vehicles require a reduction in the size and weight of power electronic modules; to meet these needs, high-dielectric-constant thin-film ceramic capacitors are being developed.

Since the previous review in 1999, several changes have occurred in the Section's staff. Two new postdoctoral fellows (Jee and Li), and two permanent staff members (Ma and Lee) were hired to work on various programs. Eight of our Laboratory Graduate Participants received graduate degrees and took positions in industry or academia. One staff member joined another section (Tribology) in the ET Division and one retired.

Our facilities have been upgraded by expansion of our laboratory space and several purchases. New pulsed laser deposition (PLD) and cryostat units were purchased for superconductor work. A liquid delivery system was installed in our metalorganic chemical

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vapor deposition (MOCVD) unit to carry out thin-film capacitor research. A gas chromatograph (GC)/mass spectroscopy (MS) was purchased for ceramic membrane research. Several new pieces of mixing equipment and furnaces were obtained for our work on phosphate ceramics.

Superconductor Development

Superconductor development remains the largest program in the Ceramics Section. The goal of this program is to develop ceramic superconductors for applications in the generation, storage, transmission, and distribution of electrical energy. The DOE program target is to produce, in collaboration with industrial partners, long lengths (≈ 500 m) of conductor capable of carrying 10^5 – 10^6 A/cm² at temperatures > 35 K. Program focus is on the two systems that currently appear to be the most promising as practical engineering materials: Y-Ba-Cu-O (YBCO) and Bi(Pb)-Sr-Ca-Cu-O (BSCCO). However, to maintain its position in the forefront of high- T_c superconductor research, the program also investigates promising new materials as they are developed.

To be used in electric power applications, superconductors must have high critical current density (J_c) and, equally important, they must be available in long lengths. Powder-in-tube (PIT) processing of BSCCO materials remains the most promising method for fabricating long superconductors with high J_c . In PIT processing, the precursor powder is sealed in a silver tube, then mechanically worked and heat treated to form a thin tape of silver (or silver alloy)-sheathed superconductor.

The Section collaborates with two world leaders in PIT processing of BSCCO superconductors: American Superconductor Corporation (ASC) and Intermagnetics General Corporation (IGC). In the work with ASC, the focus is on phase formation, reaction kinetics and optimization of the microstructure and properties of tapes. As part of this collaboration, effects of stoichiometry and heat-treatment conditions on phase-evolution kinetics are studied. Our agreement with ASC involves interactions with the University of Wisconsin, Oak Ridge, and Los Alamos National Laboratories. Our findings are incorporated into ASC's tape manufacturing scheme, and ASC recently reported a world-record J_c of 70 kA/cm² in a short rolled tape. ASC manufactured ≈ 400 km of BSCCO tapes in 2000. Individual pieces were ≈ 160 m in length and had $J_c \approx 50$ kA/cm² (average critical current ≈ 120 A) at 77 K. The collaboration with IGC is directed chiefly at issues associated with fabricating long lengths of superconductors. It has resulted in fabrication of 75 km of tapes since the previous review. The long-length (> 200 -m) tapes carry a critical current of ≈ 35 A at 77 K. From such a conductor, the world's largest high- T_c coils (1 m diameter and 0.75 m tall) were constructed for a current-limiter application. More than 45 km of tape was used to construct this coil.

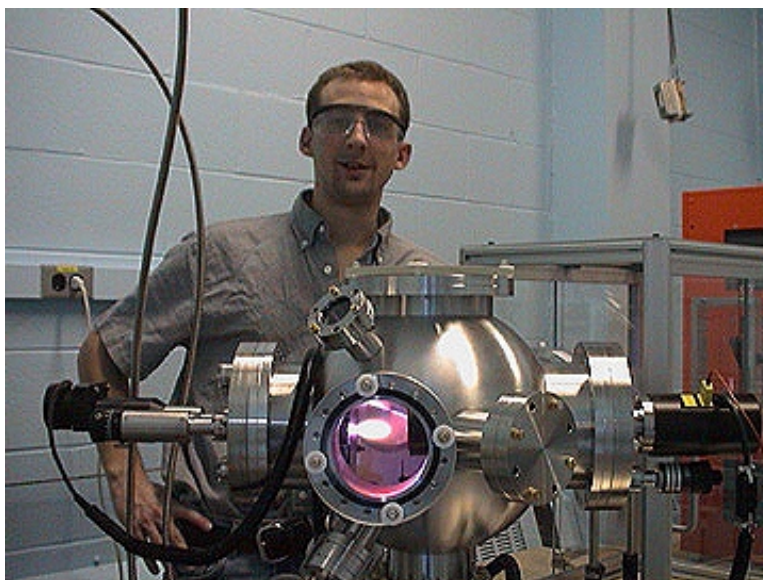
To characterize the superconducting properties of long-length conductors, a test facility was assembled. This facility allows us to pass > 2000 A of current through 1-m-long bulk superconductor rods and tubes. Performance of superconductors in applied background magnetic field (an important parameter for practical application) is also determined in our test facility.

Because BSCCO exhibits poor flux pinning (i.e., less resistance to the motion of magnetic flux) characteristics, applications of this superconductor have been limited to those areas where operations are conducted at 35 K and in a magnetic field of 1 T, or at 65 K

in a field of ≈ 0.1 T. Because YBCO behaves better than BSCCO in the presence of a magnetic field, efforts are now underway to use it for conductor development. Fabrication of well-aligned YBCO by the PIT technique is very difficult, so an alternative approach for depositing YBCO on a textured surface was developed; this is usually referred to as the “coated-conductor” method. Close control of substrate/buffer layer(s) texture is required for production of useful coated conductors. We are working on two methods to fabricate textured substrates for YBCO conductors: ion-beam-assisted deposition (IBAD) of yttria-stabilized zirconia (YSZ), and inclined substrate deposition (ISD) of magnesium oxide and other ceramics.

The section collaborates with ASC, IGC, and Universal Energy Systems, Inc. (UES) in developing coated conductors. In the work with ASC, the focus is on ISD and electrical, mechanical and microstructural characterization of coated conductors. The collaboration with IGC is directed at issues associated with IBAD, PLD, and MOCVD processes. Substrates prepared by ISD are also provided to IGC. Work with UES focuses on development of PLD technique to deposit YBCO on substrates prepared at UES.

In the IBAD approach, biaxially textured YSZ (≈ 1 μm thick) is deposited on polished, flexible Inconel or Hastelloy substrates by evaporating an yttria-stabilized zirconia (YSZ) source with an e-beam and aligning the YSZ in the preferred direction with the help of an Ar-ion source. YBCO deposited by PLD as shown in the figure below exhibited a J_c of 1.2×10^6 A/cm² at 77 K in self-field. In a field of 5 T, J_c of 1.5×10^5 A/cm² and 6×10^4 A/cm² were obtained when applied parallel to ab-plane and c-axis, respectively. Effort is also focused on developing nonvacuum processes such as sol-gel, MOCVD, and metalorganic deposition (MOD) technique for the superconductor and buffer layers. ANL’s Advanced Photon Source is used to characterize the coated conductor samples. In the ISD approach, a flexible metallic substrate is held at an angle to the direction of the plume of the species to be deposited, and texturing is obtained without the assistance of an ion gun. We have evaporated MgO by e-beam and obtained biaxially textured MgO layers on Hastelloy substrates. The ISD deposition rate is more than one order of magnitude faster than in IBAD. After depositing the appropriate buffer layers, YBCO will be deposited on ISD samples.



PLD system for deposition of superconductor and buffer layers

Many of the envisioned applications of high-temperature superconductors require greater resistance to the motion of magnetic flux, i.e., improved flux pinning. To improve flux pinning, nanophase oxides such as TiO_2 , ZrO_2 , and Al_2O_3 were added to $\text{YBa}_2\text{Cu}_3\text{O}_x$, $\text{Bi}_2\text{Sr}_2\text{CaCu}_2\text{O}_x$, and $\text{TlBa}_2\text{Ca}_2\text{Cu}_3\text{O}_x$. These additions resulted in formation of nanometer-scale compounds such as BaTiO_3 and BaAl_2O_4 and increased pinning by factors of 2 to 10 at temperatures below 50 K. In another approach, sol-gel methods were used to directly form intragranular nanophase inclusions during the synthesis reaction. We are attempting to synthesize the Bi-1212 compound to study its superconducting properties. We believe that its irreversibility field may be superior to that of Bi-2223 due to the smaller separation between Cu-O planes.

To design optimal superconductor processing protocols and final components, a large body of fundamental engineering data is required. We have produced dense, highly textured and phase-pure BSCCO bars for use in property characterization. Recent studies include measurement of the elastic-constant tensor, high-temperature mechanical properties, chemical diffusion of oxygen, and the effect of applied magnetic field magnitude and direction on transport critical current density.

Ceramic Membrane Development

The Ceramics Section is developing dense ceramic membranes for separating hydrogen from gas mixtures as part of the effort by the DOE Office of Fossil Energy (FE) to maximize the use of vast domestic fossil resources and ensure a fuel-diverse energy sector while responding to global environmental concerns. The development of cost-effective membrane-based reactor and separation technologies is of considerable interest for applications in advanced coal-based power and fuel technologies. Because concerns over global climate change are driving nations to reduce CO_2 emissions, hydrogen is considered the fuel of choice for both electric power and transportation industries. While it is likely that renewable energy sources will ultimately be used to generate hydrogen, fossil-based technologies will be utilized in the interim to generate hydrogen.

Petroleum refineries currently employ cryogenics, pressure swing adsorption (PSA), and membrane systems for hydrogen recovery. Each of these technologies has limitations: cryogenics is generally used only in large-scale facilities with liquid hydrocarbon recovery, because of its high capital cost; and PSA typically recovers less of the feedstream hydrogen and is limited to modest temperatures. Currently used membrane systems are susceptible to chemical damage from H_2S and aromatics and have limited temperature tolerance. Dense ceramic membranes made from proton-conducting materials are simple and efficient alternatives to the existing methods for hydrogen recovery.

In developing dense ceramic membranes that efficiently and economically recover hydrogen, we surveyed the literature on mixed electronic/protonic conductors to identify suitable candidate materials. $\text{SrCe}_{1-x}\text{M}_x\text{O}_{3-\delta}$ and $\text{BaCe}_{1-x}\text{M}_x\text{O}_{3-\delta}$ (where M is a fixed-valent dopant such as Ca, Y, Yb, In, Nd, or Gd) were selected for further investigation on the basis of their reported total conductivities and proton transference numbers. The transport properties of the various compositions were investigated by impedance spectroscopy, open-cell voltage (OCV), and gas permeation measurements. Because the results indicated that the electronic conductivity was insufficient for nongalvanic hydrogen separation, cermet

membranes containing $\text{BaCe}_{1-x}\text{Y}_x\text{O}_{3-\delta}$ were fabricated with increased electronic conductivity. We have demonstrated the proof of principle of separating hydrogen from gaseous mixtures by operating the membranes in the nongalvanic mode in the temperature range of 600-900°C. A GC setup, as shown in the figure below, is used to measure hydrogen permeation.



Hydrogen permeation measurement with GC setup

Currently a three-pronged approach is followed to develop suitable membrane materials:

- (a) Monolithic mixed-conducting ceramics (e.g., zirconates, Eu-doped cerates, perovskites doped on both A and B sites, etc.).
- (b) Cermets consisting of mixed electronic/protonic-conducting ceramics and a metallic component. In these cermets, the metallic component may have low hydrogen permeability, in which case the metal enhances the hydrogen permeability of the ceramic phase by increasing the electronic conductivity of the composite. Alternatively, the metallic component may have high hydrogen permeability, in which case it enhances the hydrogen permeability of the ceramic, but a significant fraction of the hydrogen passes through the metal itself.
- and (c) Composites consisting of a suitable matrix material and a metal with high hydrogen permeability. In these composites, hydrogen is transported almost exclusively by the metal phase, and the matrix serves solely as a structural support for the metal phase. Hydrogen flux of $> 3 \text{ cm}^3 (\text{STP}) \cdot \text{min}^{-1} \cdot \text{cm}^{-2}$ has been measured through ≈ 0.3 -mm-thick membranes at 900°C. Methods for fabricating these materials into thin, dense membranes are being developed, and the chemical and mechanical stabilities of the membranes are being studied to estimate their expected lifetime. Scoping-level evaluations will be performed to identify potential applications of proton membrane technology. Areas that will be evaluated include overall market scale, typical site operating scale, process integration opportunities and issues, and alternative-source economics. The Section is working with Eltron Research, Inc., Praxair, and ITN Energy Systems, Inc., in the area of hydrogen transport membranes.

The largest quantities of H_2 are currently produced from “syngas” (short for synthesis gas, a mixture of CO and H_2). Syngas is produced by steam reforming of methane or natural gas, but this approach is both energy- and capital-intensive. As a cost-effective alternative to present-day approaches, the Ceramics Section has developed ceramic membranes that allow air to be used in syngas-generation reactions. The ceramic separates oxygen from air, and the oxygen can then be used for partial oxidation of the methane (the chief constituent of natural gas) to form syngas. In principle, air flows on one side of the membrane while methane flows on the other side. Because the membrane is permeable to oxygen, but not to nitrogen, the oxygen diffuses through the membrane and reacts with the methane to generate the product gas. No external electrical source or circuitry is required because the membrane is a mixed ionic/electronic conductor. Maintaining the mechanical integrity of the membrane under the severe reactor operating conditions required that we develop a new material based on the Sr-Fe-Co-O system.

Reactor tubes extruded from the new Sr-Fe-Co-O composition have operated without failure for >1000 h at $\approx 900^\circ\text{C}$ with a methane conversion efficiency of >98%. The syngas created by partial oxidation of methane produces two moles of hydrogen per mole of methane converted. By reacting syngas with steam (in the water gas shift reaction or WGSR) in a second reactor under conditions controlled to maximize hydrogen production, we have increased the production of H_2 to ≈ 2.9 moles per mole of methane converted. These results are in excellent agreement with the thermodynamic prediction. Future efforts will focus on improvements in the membrane and seals for better performance. In addition, partial oxidation of methane by oxygen (exothermic reaction) and steam reforming (endothermic reaction) can be combined in conjunction with the WGSR to increase the process efficiency and hence maximize the economical production of hydrogen.

Waste Stabilization

The Ceramics Section has demonstrated a process for stabilizing hazardous, radioactive, and mixed wastes by using novel phosphate ceramics that set at room temperature. Chemically bonded ceramics based on magnesium, magnesium/potassium, iron, and zirconium phosphates can stabilize a wide range of DOE waste streams, such as contaminated ash, salt cakes, and sludge, and thus they hold promise for treating waste streams generated by the utility, chemical, and defense industries. The process stabilizes problem wastes such as secondary waste streams arising from thermal treatment processes (such as vitrification and plasma arc treatment) and other chemical processes used for destroying organics. The process also can be used to stabilize wastes containing volatile species and pyrophorics, which cannot be treated by thermal stabilization.

Stabilization of these materials involves both chemical solidification and physical encapsulation of wastes, resulting in superior waste forms. Optimization of the phase compositions, microstructures, and mechanical and physical properties has led to the development of dense and durable waste forms. Because of the high waste loading and low-temperature operation, chemically bonded phosphates are a low-cost alternative to other stabilization technologies. As part of treatability studies, several mixed wastes from DOE facilities such as Savannah River Site, Fernald, Rocky Flats, Argonne East and West, and Idaho National Engineering and Environmental Laboratory have been successfully stabilized in phosphate ceramics. The process has been scaled up to stabilize actual wastes;

collaborations with private industry are ongoing to demonstrate the stabilization of DOE waste streams.

Chemically bonded phosphate ceramics have been shown to bind various high-volume innocuous wastes such as plastics, cellulose fibers, ash, and lumber. With our phosphate ceramics technology, these wastes can be made into inexpensive value-added construction products. The waste materials are mixed in powder or shredded form with the phosphate binder and fabricated into a variety of forms such as blowable or pumpable insulation, particle boards, and bricks. Because the phosphate binder is inorganic, the resulting products are nonflammable. Relevant properties of all resulting materials have been evaluated to establish their applicability for storage or for lightweight, fire-resistant, environmentally benign construction materials for energy conservation. The phosphate-bonded ceramic technology has found potential application as a root canal cement, and we have started working with a company to modify the ceramic composition to make it biocompatible. Recently, the phosphate ceramic technology has been licensed to five companies for use in waste management and structural applications.

Structural Ceramics and Composites

A new project focuses on development of ceramic fibrous monoliths (FMs) for primarily structural applications. FMs are very tough and exhibit graceful failure, although their strengths are generally lower than those of continuous-fiber ceramic composites (CFCCs). They can be inexpensively produced in a wide variety of forms by conventional processing methods such as extrusion. To fulfill the promise of FMs, work is needed to tailor constituent properties and optimize design methodologies. Particular emphasis is placed on oxide systems because of their inherent stabilities at high temperature. The project involves investigations to (a) develop FMs that can be pressureless-sintered rather than hot pressed, (b) develop continuous extrusion technologies to replace the current batch processes now used in industry, (c) evaluate the performance of current FMs and develop micromechanical models to guide design of new FMs and predict their properties, and (d) develop collaborations with industry and produce useful prototype parts.

We have fabricated zircon-based FMs and determined that as with CFCCs, they are relatively insensitive to the presence of flaws. We have produced continuously extruded filament and are examining its use in solid freeform fabrication of FMs. We are working on improved FMs based on mullite and alumina. We have established effective collaborations with the leading manufacturer of FMs, Advanced Ceramics Research (Tucson, AZ), and the Universities of Illinois at Urbana-Champaign, California at Santa Barbara, and Missouri at Rolla.

The Ceramics Section has been involved in testing and developing advanced refractory materials for the iron and steel industries. For many years, we have been working with Magneco/Metrel and Answer Technology to develop and test improved refractories for various applications (torpedo and iron mixing ladles, reheat furnace hearths, and subhearths of steel processing, etc.) in iron- and steelmaking.

In a related DOE-funded program, we evaluate candidate refractories and develop refractory repair materials with improved performance and service life for use in electric arc

furnaces. This is a collaborative effort between steel and refractory producers, the University of Alabama and Clemson University, and national laboratories. Based on input from participating steel producers, key processing and testing parameters are being identified, and refractories with controlled composition and microstructures are being fabricated by participating refractory producers. These refractories are being tested and evaluated at Argonne for phase composition, microstructure, corrosion/erosion resistance, and performance in simulated service environments. Corrosion properties, microstructure, and service performance are correlated to establish refractory wear mechanisms and to develop refractory composition and microstructures with improved wear resistance and service life.

We have been involved in a DOE-funded program on thermal barrier coatings (TBCs). This work is in support of DOE's Advanced Turbine System (ATS) Program, which is aimed at developing a new generation of land-based gas turbine systems that provide cost-effective utility and industrial power generation at low cost, improved efficiency, and increased service life, and that meets environmental requirements. Our effort has been directed toward providing critical information on processing, microstructure, residual stresses, mechanical/thermomechanical properties and failure modes/mechanisms in order to develop improved TBCs for ATS applications. To this end, we have established a test methodology/protocol to measure elastic modulus of thin TBCs based on indentation techniques. This technique is very quick and cost-effective and is now routinely used to monitor TBC damage in simulated service environments. A bottom-loading furnace has been set up to perform thermal cyclic testing of TBCs in oxidizing environment, and a mechanical test facility attached with a high-power microscope to monitor in-situ damage evolution has been built for mechanical fatigue experiments. In a parallel effort, we have established test procedures that use ruby fluorescence spectroscopy to measure residual stresses in TBC systems. These stresses are critically needed for damage evolution and lifetime prediction modeling.

Capacitor Development

The Ceramics Section is developing advanced capacitor technologies to support DOE's program on vehicle electrification. Advanced power electronic modules, the heart of the electrical propulsion system, require low-cost, high-energy-density capacitors with reduced weight and size. Such capacitors can be realized by fabrication of microelectronic-scale ($\text{Ba}_{1-x}\text{Sr}_x$) TiO_3 (BST) thin-film ($<0.5\ \mu\text{m}$) capacitors. Vapor-deposited BST, a ferroelectric material, possesses superior dielectric properties such as high dielectric constants, high breakdown fields, and low losses.

Our efforts are centered on fabricating BST thin films by MOCVD, developing a fundamental understanding of processing effects on dielectric properties, and arriving at robust processing strategies for anticipated devices. This program is a collaborative effort with ANL's Materials Science Division.

We have designed a novel large-area MOCVD reactor and developed robust processing methodologies to fabricate high-quality BST thin films. The MOCVD reactor, as shown in the next figure, includes a well-controlled vapor delivery path and substrate heater, and a commercial liquid precursor chemical delivery tool (ATMI LDS-300B) to allow strict control of the film composition. A feedback metric has been designed to evaluate growth efficiency of



MOCVD system for depositing BST films

the thin films. As a result, BST films with excellent compositional and thickness uniformity across a 4 in. wafer, together with growth rates as high as 120 Å/min with good reproducibility, have been obtained.

The effects of film composition, growth conditions, and postprocessing annealing were investigated and correlated with electrical properties. Appropriate growth conditions were determined for deposition of uniaxially oriented polycrystalline BST thin films, a chief advantage of the MOCVD process. Dielectric constants of 580 at zero bias, dielectric losses as low as 0.003, and breakdown fields of 2.5 MV/cm have been obtained. Such properties lead to energy densities as high as 21 J/cm³ for the dielectric film. Higher energy densities are anticipated with continued process development.

Concurrently with this effort, we are investigating the replacement of cost-prohibitive noble metal capacitor electrodes with more economical base metals. An additional effort is directed at developing ferroelectric thin films for advanced microwave communication technologies. Argonne works closely with the Center for Dielectric Studies at Pennsylvania State University and the University of California at Santa Barbara. We have also developed strong working relationships with microelectronic equipment companies developing ferroelectric thin film technologies, capacitor companies, and the automotive end-users.

Recent Work by the Ceramics Section

Publications

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U. Balachandran, R. Siegel, and T. Askew

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Corrosion and Mechanics of Materials

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Programs of the Corrosion and Mechanics of Materials Section address specific concerns related to corrosion and the effects of various environments on the mechanical behavior of materials used in several types of energy systems. The research is sponsored by various branches of the U.S. Department of Energy (DOE) that include the Office of Fusion Science, the Office of Fossil Energy, and the Office of Industrial Technologies. The light water reactor (LWR) work, which includes studies of effects of reactor environments on low-cycle fatigue and crack propagation in reactor structural alloys, irradiation-induced susceptibility to stress corrosion cracking, and cladding criteria for high-burnup fuel, is supported by the U.S. Nuclear Regulatory Commission (NRC). Research on aqueous, gaseous, and liquid-metal corrosion, thermodynamics and kinetics of gas/solid reactions, and high-temperature mechanical properties in four major program areas is summarized below.

Light Water Reactors

To continue safe operation of current LWRs, the aging degradation of the reactor structures must be adequately understood and managed. Potential aging mechanisms include fatigue and environmentally assisted cracking (EAC) of piping and pressure vessels, and irradiation assisted stress corrosion cracking of reactor internals. Nonsensitized austenitic stainless steels (SSs) become susceptible to intergranular failure after accumulation of a sufficient neutron fluence. Such cracking has occurred in control-blade sheaths and handles and in instrument dry tubes of boiling water reactors (BWRs). Intergranular cracking has also occurred in more safety-significant structural core components in BWRs, such as the top guide, shroud, and core plate. However, the relative contributions of neutron fluence, material composition, heat-treatment condition (sensitization), and fabrication variables (welding method and residual weld and fit-up stresses) to crack initiation and growth are not clear. The

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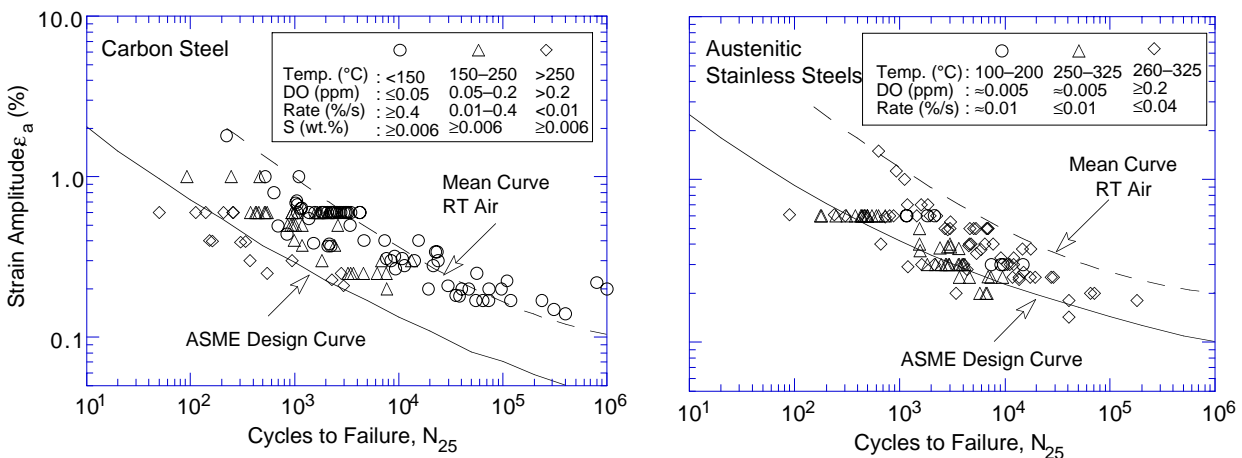
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current research on EAC of LWR materials has focused on (a) fatigue of pressure vessel and piping steels, (b) crack growth in austenitic SSs, (c) IASCC of austenitic SSs, and (d) EAC in high-nickel alloys.

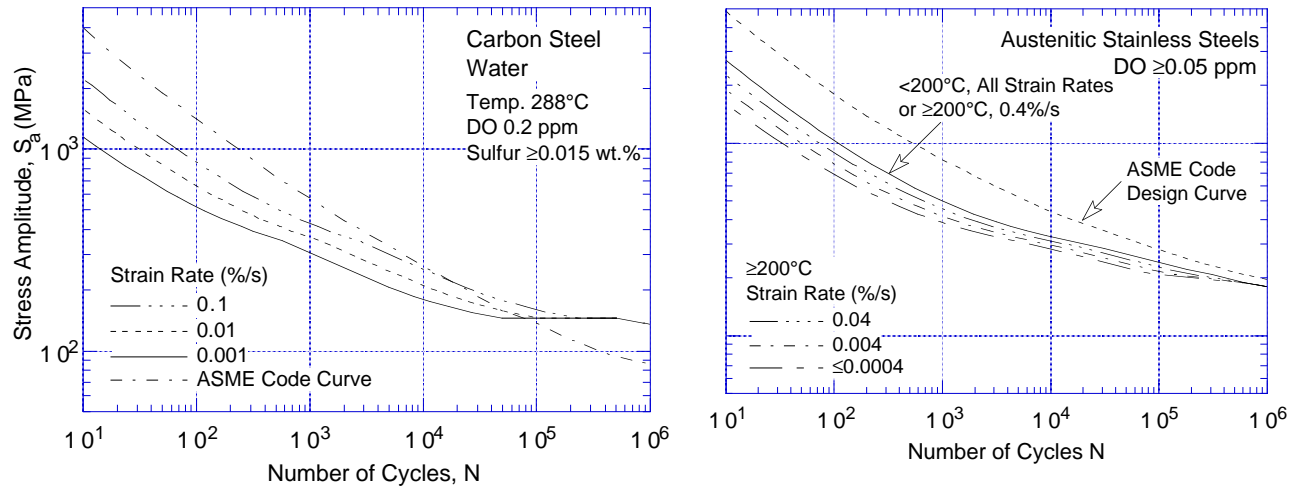
Fatigue Testing of Carbon Steels and Low-Alloy Steels in Simulated Reactor Coolant Environments

Carbon and low-alloy steels and austenitic stainless steels are used extensively in LWR steam supply systems as piping and pressure vessel materials. The environmental enhancement of fatigue crack growth rates in pressure vessel and piping steels in high-temperature water is well known. LWR coolant environments can also have significant effects on the fatigue lives of these steels. Under certain conditions of loading and environment, the fatigue lives of these steels, shown in the figure below, can be a factor of 70 lower in coolant environments than in air. Fatigue tests are being conducted in air and simulated LWR environments to obtain data under conditions where information is lacking in the existing fatigue S-N data base. Additional studies have been undertaken to determine crack initiation and crack growth characteristics, and to better understand the actual mechanism of degradation. The results indicate that the decrease in fatigue life of pressure vessel and piping steels is caused primarily by the effect of environment during the early stages of fatigue damage, i.e., growth of cracks that are <200 μm deep (Stage I crack growth). The mechanisms for the enhancement of the growth rates are slip dissolution/oxidation in carbon and low-alloy steels and hydrogen-induced cracking in austenitic stainless steels.



Fatigue S-N data for carbon steel and austenitic stainless steels in water

Two approaches have been proposed for incorporating the effects of LWR environments into ASME Section III fatigue evaluations: (a) development of new design fatigue curves for LWR applications, and (b) use of a fatigue life correction factor to account for environmental effects. Both of these approaches are based on the existing fatigue S-N data in LWR environments, i.e., the best-fit curves to the experimental fatigue S-N data in LWR environments are used to obtain the design curves or the fatigue life correction factor. Environmentally adjusted design fatigue curves, shown in the next figure, have been developed from the best-fit curves to the experimental data in LWR environments by the same procedure used to develop the current ASME Code design fatigue curves. The best-fit experimental curves are first adjusted for the



Environmentally adjusted design fatigue curves developed from experimental data for carbon steel and austenitic stainless steels in LWR environments

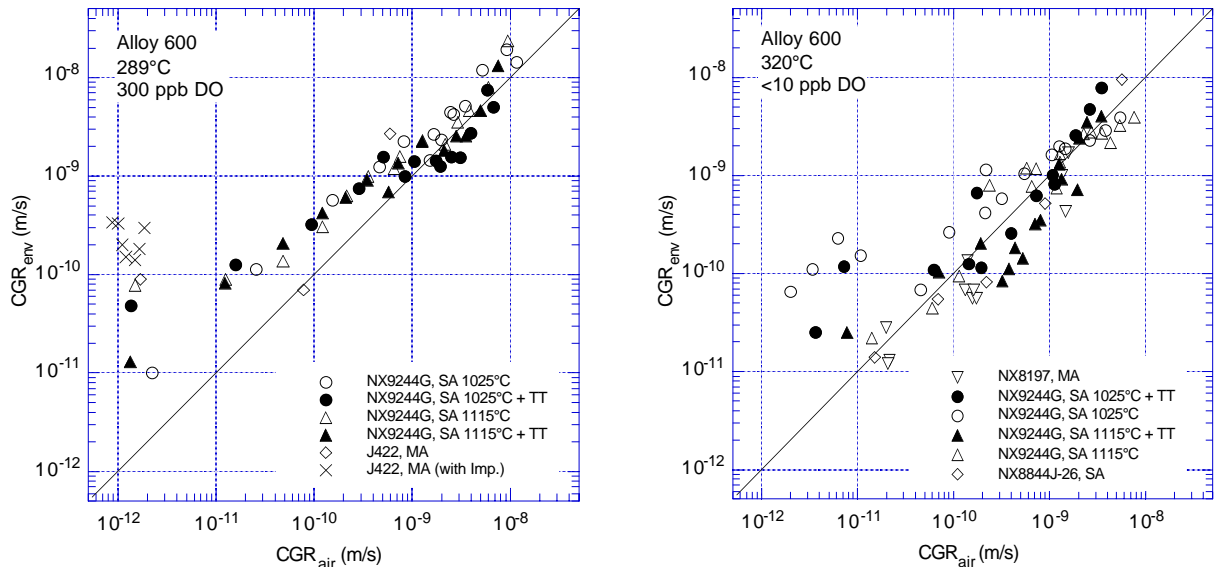
effect of mean stress by using the modified Goodman relation. The design fatigue curves are then obtained by lowering the adjusted best-fit curve by a factor of 2 on stress or 20 on cycles, whichever is more conservative, to account for differences and uncertainties in fatigue life associated with material and loading conditions.

The effects of reactor coolant environments on fatigue life have also been expressed in terms of a fatigue life correction factor F_{en} , which is the ratio of the life in air to that in water. A nonmandatory appendix based on this procedure is being proposed for inclusion in Section III of the ASME Code. To incorporate environmental effects into the Section III fatigue evaluation, the fatigue usage for a specific stress cycle based on the current Code design fatigue curve is multiplied by the correction factor.

Environmentally Assisted Cracking of Alloys 600 and 690 in LWR Coolant Environments

Alloys 600 and 690 are used for a variety of structural elements in reactor systems in addition to their use as steam generator tubing. Cracking has been observed in a number of components, e.g., instrument nozzles and heater thermal sleeves in the pressurizer, penetrations for control-rod-drive mechanisms in reactor vessel closure heads in the primary system of PWRs, and in shroud-support-access-hole covers in BWRs. Experience strongly suggests that materials that are susceptible to SCC are also susceptible to environmental degradation of fatigue life and fatigue-crack-growth properties. Information has been obtained on the effect of temperature, load ratio, and stress intensity on EAC of these alloys in simulated BWR and PWR water, and the crack growth rates (CGRs) of these materials have been compared with rates determined in air.

To obtain a qualitative understanding of the degree of enhancement and the range of conditions over which significant environmental enhancement is observed, it is helpful to plot the observed CGRs against the CGRs that would be expected in air under the same mechanical loading conditions, i.e., the same stress intensity range, cyclic stress ratio, and rise time. The next figure shows corrosion fatigue results for various heats of Alloy 600 tested in various heat



Corrosion fatigue data for Alloy 600 in high-dissolved-oxygen (≈ 300 ppb) water at 289°C and low-dissolved-oxygen (< 10 ppb) water at 320°C

treatment conditions. There is significant environmental enhancement of the CGR at both low- and high-DO levels. For CGRs greater than $\approx 10^{-9}$ m·s⁻¹, the mechanical driving forces dominate; at lower CGRs, the environmental contributions dominate.

Irradiation-Induced Stress Corrosion Cracking of Austenitic Stainless Steels

In recent years, failures of reactor internal components have been observed after the components have reached neutron fluence levels $> 5 \times 10^{20}$ n·cm⁻², $E > 1$ MeV. The general pattern of the observed failures indicates that as nuclear plants age and fluence increases, various apparently nonsensitized austenitic stainless steels become susceptible to intergranular failure. Some components (e.g., BWR core shroud, control-blade handle and sheath) have cracked under low applied stresses. Although some failed components can be replaced, structural components such as the BWR top guide, shroud, or core plate would be very difficult or impractical to replace. The understanding of this type of degradation, which is commonly known as irradiation-induced stress corrosion cracking (IASCC), is necessary to determine operating and inspection requirements for reactors that have reached threshold fluence levels.

The primary effects of irradiation on the reactor internals, which are usually fabricated from ASTM Type 304, 316, or 348 SSs, include alteration of microchemistry, microstructure, and mechanical properties. Irradiation produces defects and defect clusters in grain matrices and alters the dislocation network, dislocation loop, and dislocation channel structures, leading to radiation-induced hardening. Irradiation also leads to changes in the stability of second-phase precipitates and the local chemistry near grain boundaries, precipitates, and defect clusters. Grain-boundary microchemistries that differ significantly from that of the bulk composition can be produced not only by radiation-induced segregation (RIS) but also by

thermally driven equilibrium and nonequilibrium segregation of alloying and impurity elements. Neutron irradiation also alters the water chemistry.

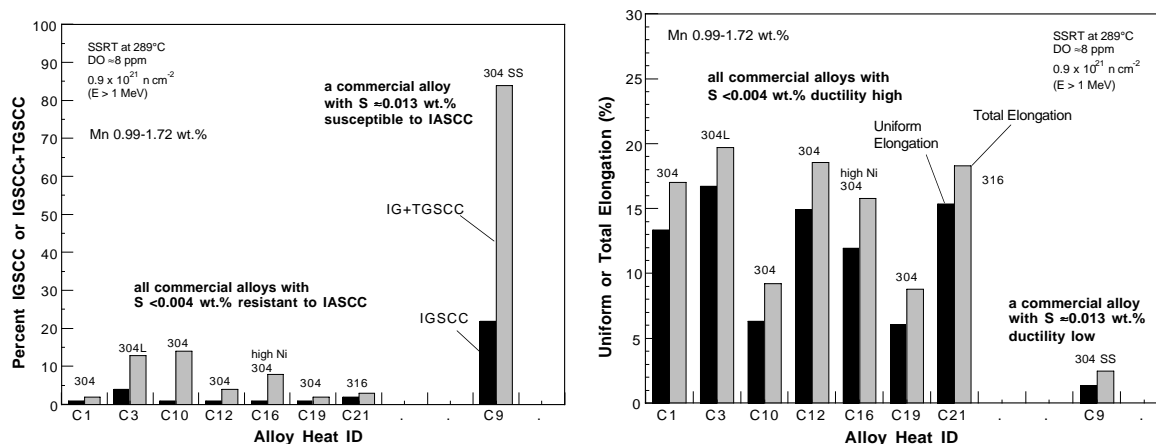
For many years, irradiation-induced grain-boundary depletion of chromium has been considered to be the primary metallurgical process that causes IASCC. One of the most important pieces of evidence in support of the chromium-depletion mechanism is the similar dependence of intergranular stress corrosion cracking (IGSCC) of thermally sensitized material and of IASCC of BWR-irradiated solution-annealed material on water chemistry (i.e., oxidizing potential) is similar. Recently, attention has also been given to the effects of fabrication-related variables, i.e., grain-boundary segregation and depletion of alloying and impurity elements by thermal processes, the effect of final thermomechanical treatment, hardening by cold work, the use of recycled scrap metals, uptake of minor impurities during iron- and steelmaking, and contamination by minor impurities during welding of field components such as BWR core shrouds.

Our work in this area includes (a) slow-strain-rate-tensile testing of model stainless steel alloys irradiated in Norway's Halden heavy-water-boiling reactor in BWR-like water; (b) stress corrosion testing, tensile testing, and microstructural analysis of decommissioned EBR-II components irradiated to very high dose; (c) microstructural characterization and failure analyses of BWR core shroud welds and simulated mockup welds fabricated by shielded-metal-arc procedure; (d) analysis of welding-related impurity contamination and microstructural evolution in field-cracked Type 304 SS core shroud weld by shielded secondary ion mass spectroscopy (SIMS) in cooperation with the Paul Scherrer Institute in Switzerland; and (e) test design and specimen fabrication for a PWR-relevant irradiation experiment in the Russian BOR-60 Reactor in cooperation with the International Group on Cooperative IASCC Research. Progress and highlights of these efforts are summarized below.

IASCC of Model Austenitic Stainless Steels

In this task, slow-strain-rate tensile tests in simulated BWR water were conducted on model austenitic stainless steel alloys that were irradiated at 289°C in helium to $\approx 0.3 \times 10^{21}$ n·cm⁻² and $\approx 0.9 \times 10^{21}$ n·cm⁻² ($E > 1$ MeV) in the Halden reactor. Fractographic analysis by scanning electron microscopy was conducted to determine susceptibility to IASCC as manifested by the degree of intergranular and transgranular stress corrosion cracking (IGSCC and TGSCC) fracture surface morphology. As fluence was increased from $\approx 0.3 \times 10^{21}$ n·cm⁻² to $\approx 0.9 \times 10^{21}$ n·cm⁻², IGSCC fracture surfaces emerged in many alloys, usually in the middle of and surrounded by TGSCC fracture surfaces. Alloy-to-alloy variations in susceptibility to TGSCC and IGSCC were significant at $\approx 0.9 \times 10^{21}$ n·cm⁻². Susceptibility to TGSCC and IGSCC was influenced by more than one alloying and impurity element in a complex manner.

Results from this study indicate that for commercial heats of Types 304 and 304L SS, S concentrations of >0.013 wt.% are detrimental and that a sufficiently low concentration of S (<0.004 wt.% S) is a necessary condition for good resistance to IASCC (see next figure). A laboratory alloy containing a high concentration of Cr (21 wt.% Cr) and <2 vol.% of delta ferrite exhibited excellent resistance to TGSCC and IGSCC, despite a high S content, thus confirming that Cr in high concentrations plays a major role in suppressing susceptibility to IASCC under BWR conditions. The yield strength of the model alloys was nearly constant at ≈ 200 MPa in the unirradiated state and was generally independent of Si concentration. However, as the alloys



Effects of S on susceptibility to TGSCC and IGSCC (left) and uniform and total elongations (right) of Type 304 SS measured after irradiation to $\approx 0.9 \times 10^{21} \text{ n cm}^{-2}$ ($E > 1 \text{ MeV}$)

were irradiated, the degree of increase in yield strength was significantly lower for alloys that contain $>0.9 \text{ wt.}\%$ Si than for alloys that contain $<0.8 \text{ wt.}\%$ Si, indicating that the nature of irradiation-induced hardening centers and the degree of irradiation hardening is significantly influenced by the Si content of the alloy. Similar effects were not observed for C and N. Results also indicate that a Si content between ≈ 0.9 and $\approx 1.5 \text{ wt.}\%$ is beneficial in delaying the onset of or suppressing the susceptibility to IASCC. Although susceptibility to TGSCC and IGSCC was insignificant and the fracture surface morphology was mostly ductile, some alloys exhibited very low uniform elongation and poor work-hardening capability in water after irradiation to $\approx 0.9 \times 10^{21} \text{ n cm}^{-2}$. Such alloys contained unusually high concentrations of O ($>0.02 \text{ wt.}\%$ O) or unusually low concentrations of Si ($<0.4 \text{ wt.}\%$ Si) or both.

IASCC of Austenitic Stainless Steel Irradiated to Very High Dose

Some core internal structural components of LWRs, typically fabricated from Types 304, 316, or 347 austenitic stainless steel, accumulate very high levels of irradiation damage (i.e., up to 18 and 80 dpa, respectively, in BWR and PWR) by the end of life. Under life-extension situations (e.g., life extended from 40 to 60 yr), even higher damage levels will be accumulated. Our data bases and mechanistic understanding of the degradation of such highly irradiated components, however, are not well established. A key question is the nature of irradiation-assisted intergranular cracking at very high dose, i.e., is it purely mechanical failure or is it stress-corrosion cracking?

In this task, hot-cell tests and microstructural characterization were performed on Type 304 SS from the hexagonal fuel can of the decommissioned EBR-II reactor after irradiation to $\approx 50 \text{ dpa}$ at $\approx 370^\circ\text{C}$. Slow-strain-rate tensile tests were conducted at 289°C in air and in water at several levels of electrochemical potential (ECP), and microstructural characteristics were analyzed by scanning and transmission electron microscopy. In air, the material deformed by twinning and exhibited surprisingly high ductility. In high-ECP water, the material was susceptible to severe IGSCC. However, low levels of dissolved O and ECP ($< -320 \text{ mV SHE}$) were effective in suppressing the susceptibility of the heavily irradiated material to IGSCC, indicating that the failure was associated with stress corrosion due to irradiation-induced grain-boundary Cr depletion, rather than purely mechanical separation of grain boundaries. However, although

IGSCC was suppressed at low ECP, the material was susceptible to dislocation channeling, and this susceptibility led to poor work-hardening capability and low ductility.

More data on highly irradiated materials will be developed through ANL participation in an international cooperative program. In the Cooperative Research on Irradiation-Assisted Stress Corrosion Cracking (CIR) Program, stainless steel specimens will be irradiated in the BOR-60 reactor located in Dimitrovgrad, Russian Federation. Postirradiation testing and microstructural investigation will be performed in ET's hot cells.

Failure Analysis of BWR Core Shroud Welds

Failure of BWR core shroud welds, usually fabricated from Types 304 and 304L austenitic stainless steels (SSs), has increased significantly in the past several years. Although BWR core shrouds are subject to relatively low neutron fluence, many vertical and horizontal welds crack by the time they accumulate the relatively low fluences of $\approx 3 \times 10^{20} \text{ n cm}^{-2}$ ($E > 1 \text{ MeV}$). At this low fluence, nonwelded base-metal components are not considered susceptible to irradiation-assisted stress corrosion cracking (IASCC), which typically occurs after a threshold fluence of $\approx 5 \times 10^{20} \text{ n cm}^{-2}$ ($E > 1 \text{ MeV}$). Because of this consideration, and in view of the understanding gained from experience and investigation of stress corrosion cracking of gas-tungsten-arc (GTA)-welded piping and laboratory-sensitized nonwelded base-metal specimens, most cases of Type 304 SS core shroud cracking have been attributed to classical IGSCC of thermally sensitized austenitic stainless steel, in which significant grain-boundary carbide precipitation occurs in the heat-affected zone (HAZ) during GTA welding or sensitizing heat treatment. Implicit to this rationale is the assumption that significant precipitation of grain-boundary carbides occurs in the HAZ of Type 304 SS core shroud welds fabricated by the shielded-metal-arc (SMA) or submerged-arc (SA) procedure rather than by GTA. However, contrary to expectations of most investigators, an increasing number of field cracking incidents have been observed in core shroud welds fabricated from low-C Type 304L SS. This observation is difficult to explain on the basis of classical IGSCC, because grain-boundary carbides do not precipitate in low-C Type 304L stainless steels during GTA welding or after thermal sensitization.

To provide a better understanding of the cracking mechanism, failure behavior and microstructural characteristics of BWR core shroud welds were analyzed in this task by analytical electron microscopy (AEM), secondary ion mass spectroscopy (SIMS), auger electron spectroscopy (AES), scanning electron microscopy (SEM), optical microscopy (OM), and chemical analysis. Field-cracked Type 304 and 304L SS core shroud welds and unirradiated mockup welds were analyzed. Analysis by SIMS, however, was limited to unirradiated mockup welds. A major finding from the studies was that the HAZ of the field-cracked core shroud and mockup welds were contaminated with high levels of O, F, and Ca, the elements that originate from air or the CaF_2 -containing weld electrode coating. Solubility of these impurities is negligible or extremely low in austenitic stainless steels, except at high temperatures when welding passes are made. Therefore, during cooling after the completion of welding passes or during long-term aging during in-reactor operation, these impurities segregate either to grain boundaries or to oxides or sulfides in the steel. This behavior was verified by the results of complementary microstructural characterization by AES, SIMS, and AEM.

Based on these observations and evidence, a mechanistic model of core shroud weld cracking has been proposed. The model is essentially based on synergism among Cr, O, and F atoms on or near grain boundaries. According to the model, the susceptibility of the HAZ in a

core shroud weld to intergranular SCC is strongly influenced by the degree of O and F contamination and segregation of these impurities to grain boundaries. Higher concentrations of O on grain boundaries are conducive to more Cr bound to O. Fluorine segregates to grain boundaries during cooling after completion of welding passes or during long-term reactor operation. Either of these conditions is conducive to higher F ion and lower Cr ion concentrations in the crack tip environment and allows F to play a strong catalytic role in dissolution of Fe at the crack tip. The presence of dense Ca(O,F) particles, especially in HAZ <1 mm away from the fusion line, exacerbates the process because the particles dissolve readily in water, thereby releasing F ions, and because they promote formation of microcavities near grain boundaries. The model also appears to provide a good explanation for fluoride-induced intergranular stress corrosion cracking of sensitized nonwelded steel, i.e., cracking of a base metal in the presence of fluoride ions in water in which significant grain-boundary carbide precipitation occurs. When F contamination is present and grain boundaries are significantly depleted of Cr by an irradiation-induced process, as in the case of the neutron absorber tubes, the model also predicts susceptibility to IG stress corrosion cracking.

Steam Generator Tube Integrity Program

Steam generator tubes, which account for more than 50% of the primary pressure boundary surface of PWRs, have experienced in-service corrosive and mechanical degradation of various forms since the beginning of PWR commercial operation in the late 1950s. Various forms of steam generator tube degradation have resulted in the plugging of well over 100,000 tubes to date around the world. In addition, 68 steam generators in 22 U.S. plants had been replaced by the end of 1998 at a cost of about \$100 to \$200 million each, and more replacements are underway or planned. Environmentally induced degradation through IGSCC and intergranular attack (IGA) is the most serious degradation process at present. This degradation commonly occurs in creviced regions at tube support plate and tube sheet locations or under sludge piles, although IGSCC has also been observed in the free span of tubes. Because of its variable and often complex morphology, this cracking can be difficult to accurately detect and size by conventional inspection techniques, and the failure pressure and leak-rate behaviors of degraded tubes are not readily predictable. In view of the changing nature of observed tube degradation, and in order to provide a more modern and flexible approach to steam generator inspection, maintenance, and repair, the NRC is developing a new regulatory guidance for steam generators. This research program has recently been initiated to address the new and complex forms of degradation observed in recent years and to support the NRC rule-making activities.

The Steam Generator Tube Integrity Program encompasses four technical tasks: (1) Assessment of Inspection Reliability, (2) Research on Inservice Inspection Technology, (3) Research on Degradation Modes and Integrity, and (4) Integration of Results, Methodology, and Technical Assessments for Current and Emerging Regulatory Issues. The first two tasks deal with nondestructive evaluation (NDE) concerns and are being performed primarily by members of ET's Sensors, Instrumentation, and NDE Section. The work in those tasks is described in that Section's chapter of this Research Summary.

The objective of Task 3 is to evaluate and experimentally validate methodologies to predict potential degradation modes, progression rates, leak/rupture behavior, failure pressures, and leak rates for steam generator tubes under normal operating and accident conditions. Two

major test facilities have been designed and built under this task and are currently being used to generate data on the failure pressures and leak rates of steam generator tubes containing flaws. The first facility is the Pressure and Leak-Rate Test Facility, which simulates prototypical steam generator operating conditions, with a maximum test temperature of 343°C (650°F), a maximum pressure of 21 MPa (3000 psi), and pressurized water flow rates of up to 1520 L/min (400 gpm). This facility has been used to determine the failure pressures and leak rates for Alloy 600 tubes containing a variety of machined flaws and laboratory-produced stress corrosion cracks. Of particular interest is a series of tests conducted under stepped pressure levels to determine the stability and leak-rate behaviors of tight, ligamented stress corrosion cracks with pinhole throughwall penetrations. It was found that ligament tearing and crack opening commonly occur at such flaws at constant pressure and temperature, even though the test temperature of 282°C (540°F) is well below the creep range for Alloy 600. This ligament tearing is accompanied by stepwise increases in leak rate.

The second major facility designed and constructed under Task 3 is the High-Pressure Test Facility. This room-temperature facility operates at a maximum pressure of 53.1 MPa (7700 psi) and a maximum leak rate of 49 L/min (13 gpm). Its relatively high operating pressure permits the testing to failure of tubes with shallower flaws than can be failed in the Pressure and Leak-Rate Test Facility described above, but its significantly lower water supply capacity limits its ability to determine leak rates or to test tubes with initially leaking flaws. The facility is much simpler to set up and operate than the elevated-temperature Pressure and Leak-Rate Test Facility, and test turnaround times are significantly shorter. It has been used to determine the failure pressures and initial leak rates of numerous steam generator tubes with a wide variety of machined and laser-cut flaws as well as laboratory-produced stress corrosion cracks. A limited number of tests on cracked steam generator tubes removed from the McGuire Nuclear Plant are planned in order to compare the observed behavior of laboratory-cracked tubes with that of tubes cracked in service.

Well-established criteria exist for predicting ligament rupture and unstable burst pressures of tubes with relatively long rectangular flaws. Some modifications of these criteria have been made for short and deep flaws, based on the tests described above. Although we can currently predict with some confidence the failure pressures of tubes with rectangular flaws, such a morphology is not characteristic of much of the cracking that is currently being observed in steam generators. Stress corrosion cracks in steam generator tubes are generally nonplanar, ligamented, and can have highly complex geometry. Procedures for predicting ligament rupture for such complex cracks, using an “equivalent rectangular crack” approach (next figure), have recently been developed. Tests conducted at ANL on steam generator tubes with laboratory-produced stress corrosion cracks have shown the usefulness of such an approach. The use of the “equivalent rectangular crack” to predict leak rates through laboratory-generated stress corrosion cracks has also proven to be promising. Additional leak rate and failure tests on tubes with stress corrosion cracks that are generated in the laboratory, as well as on pulled tubes from a retired SG, are currently being planned to further validate the approach.

Production of tubes with prototypical stress corrosion cracks is critical to the above activities, as well as to the related NDE work being conducted under Tasks 1 and 2. Techniques for producing such cracks in the laboratory have been developed, and these techniques are being continually refined. Currently, cracked tubes are being produced by a procedure in which the tubes are exposed to an aqueous solution of 0.1-1N sodium tetrathionate at room

temperature and atmospheric pressure under load. Crack lengths, depths, and morphologies are varied by controlling the exposure and loading conditions, and a wide range of crack characteristics can be reliably produced by this technique. This approach requires that the tube first be heat treated to produce a sensitized microstructure, and this heat treatment results in an undesirable decrease in the tensile and yield strengths of the Alloy 600 tubing material. An alternative sensitization heat treatment that produces virtually no change in the mechanical properties of the tubes has recently been developed.

Experimental work is also being initiated on the influence of Pb on the stress corrosion cracking of Alloy 600. Solution pH, electrochemical potential, and Pb concentration will be the principal variables emphasized in this investigation. The development of a phenomenological model for the initiation of stress corrosion cracking on the secondary side of PWR steam generators has also begun.

Fossil Energy

Conceptual designs of advanced coal-fired combustion systems require high-temperature furnaces and heat transfer surfaces that operate at much higher temperatures than those in current coal-fired power plants. The combination of elevated temperatures and hostile combustion environments necessitates the development and application of ceramic materials in these designs. However, downstream of the combustion zone, a transition from ceramic to metallic materials will be required and the metallic components will experience much more elevated temperatures than those in current combustion systems. Furthermore, they will be subjected to combustion environments in which the deposit and gas chemistries could be different from those in current boiler systems. Studies of the high-temperature corrosion of metallic and ceramic materials are being conducted in simulated coal-gasification atmospheres and coal-combustion environments typical of conventional pulverized coal-fired boilers, fluidized-bed systems, and low-NO_x boilers and Advanced Fossil Power Systems. This effort is directed at establishing mechanisms of corrosion, predicting the onset of breakaway corrosion, and evaluating the role of stress and deformation in corrosion processes.

Sulfidation and chloridation resistance of structural alloys (namely, model Fe-Cr, Fe-Cr-Ni, Fe-Cr-Al alloys, Fe-Al intermetallics with controlled additions of several refractory metals and rare earth elements, and commercial high-Cr alloys) is being evaluated by thermogravimetric tests, and postexposure microstructure of corrosion scales is being

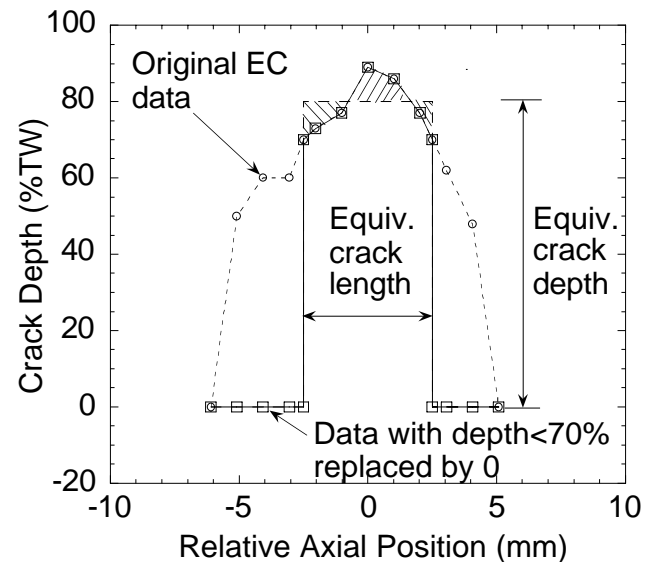
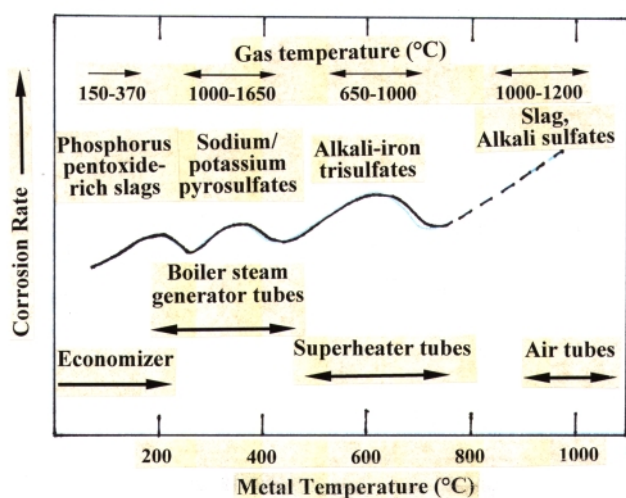


Illustration of use of "equivalent rectangular crack" to predict failure pressure of a more complex stress corrosion crack whose depth profile was obtained by eddy current (EC) techniques. More validation work on a wider variety of crack geometries is needed.

characterized by several electron/optical microscopy techniques. Alloy additions are made by bulk alloying and as overlay or diffusion coatings. Another major thrust of the program is to evaluate the effects of corrosive environments on the tensile and creep properties of metallic alloys and fracture toughness properties of monolithic and ceramic/ceramic composite materials. Further collaborative programs, supported by DOE's Fossil Energy and its Materials Division of Basic Energy Sciences, are in progress to examine fundamental aspects of scaling in structural materials exposed to oxidizing environments and to develop ultrahigh-strength Mo-Si intermetallic alloys and $\text{MoSi}_2\text{-Si}_3\text{N}_4$ composite materials.

Corrosion Behavior in Advanced Combustion Power Generation Systems

In coal-fired combustion systems, the presence of slag constituents, sulfur, alkali, and chlorine determine the thermodynamic activity of various deposit constituents. An important difference between the conventional boiler system and the advanced system is seen in the chemical and physical characteristics of the ash layers that can be deposited on the heat transfer surfaces. The adjacent figure is a schematic indication of the deposits that can lead to corrosion of waterwall boiler tubes, steam superheaters, and air tubes during service in coal-fired systems. Fireside metal wastage in conventional coal-fired boilers can occur via gas-phase oxidation or deposit-induced liquid-phase corrosion. The former can be minimized by using materials that are



Regimes of fireside corrosion in coal-fired boilers

oxidation-resistant at service temperatures of interest. On the other hand, deposit-induced corrosion of materials is an accelerated type of attack influenced by the vaporization and condensation of small amounts of impurities such as sodium, potassium, sulfur, chlorine, and vanadium, or their compounds, that are present in the coal feedstock. The deposits that form on the air tubes are likely to be dominated by alkali sulfates and coal slags in the advanced system rather than by pyrosulfates or alkali-iron-trisulfates, which form on the boiler waterwall and steam superheater materials. A major concern is the long-term performance of the air tubes because of the increased mobility of corrosion accelerating agents in the deposit layers due to the much higher temperature of the heat transfer surfaces in the advanced system.

Thermodynamic calculations were used to evaluate the chemistries of gaseous and condensed phases that occur during combustion of an Illinois bituminous coal. The computer program used for the calculations is based on calculating the equilibrium concentrations of various species by minimizing the free energy of the system. Calculated results for the chemistry of condensed phases and the gaseous phase at the combustion air:coal stoichiometric ratio of 1.3 showed that coal combustion results in a liquid phase that is essentially a silica-saturated silicate and/or sulfate condensate, with the components made up largely of SiO_2 , Al_2O_3 , CaO , Fe_2O_3 , Na_2SO_4 , and NaCl . At temperatures below 1180°C , the

partial pressures of NaCl(g) , HCl(g) , NaOH(g) , and Na(g) change abruptly; a complex sodium aluminosilicate, $\text{Na}_2\text{O} \cdot \text{Al}_2\text{O}_3 \cdot 6\text{SiO}_2\text{(s)}$, tends to form; and the liquid silicate-sulfate condensate disappears. Formation of the complex silicate requires intimate contact among several gaseous and condensed phases, but the probability of such contact in real systems is expected to be low. Under nonequilibrium conditions, where such reactions are constrained, stability of the liquid sulfate-silicate condensate extends to temperatures as low as 890°C .

Based on the results of these calculations, the general behavior of the solution phase under oxidizing conditions can be described as follows. A liquid solution phase, consisting mostly of silicates of aluminum, calcium, sodium, magnesium, and iron, is present at 1730°C . The mole fraction of Na_2SO_4 phase in the liquid is <0.01 above 1280°C . The concentration of Na_2SO_4 rises rapidly as the temperature decreases and becomes a major component of the solution at 980°C . The mole fraction of Na_2SO_4 may increase to 0.9 or more at 980°C . However, these are thermodynamic calculations and indicate only the stability of different phases that can form at various temperatures. The formation of a specific phase in practice will be strongly dictated by the kinetics; furthermore, the deposit can have a wide variation in composition and stratification of various phases, and this can also influence the corrosion performance of the underlying metal alloy. Chemistry calculations on the anticipated phases in deposits at temperatures of interest for air tubes showed mullite, Ca aluminosilicates, silica, and iron oxide when U.S. eastern coals are combusted under excess air conditions.

Several experiments were conducted to evaluate the role(s) of deposit chemistry in the corrosion performance of both metallic and ceramic materials. Results showed that three different mechanisms may be responsible for chlorine attack on structural metallic alloys. These include (a) direct reaction between structural metal constituents and Cl_2 or HCl , leading to formation of volatile chlorides; (b) reaction between oxides of structural metal constituents and Cl_2 or HCl , leading to volatile chlorides; and (c) formation of solid metal chlorides but loss of metal due to the high vapor pressure of these chlorides at the exposure temperature. Calculations were made to evaluate the equilibrium partial pressures of various volatile chloride species that form by reaction of Fe, Cr, Ni, and Al and their oxides with Cl_2 or HCl at 650°C . The calculations showed that the direct reaction of Fe, Cr, and Al with Cl_2 or HCl will result in significant pressures of volatile phases FeCl_3 , CrCl_3 , AlCl_3 , Al_2Cl_6 , and AlOCl at 650°C in the gas environments used in the present studies. Direct reaction between Ni and Cl_2 or HCl will result in the solid NiCl_2 phase with a vapor pressure of 5×10^{-4} atm at 650°C . On the other hand, reaction between Fe_2O_3 and Cl_2 or HCl would result in negligible volatile FeCl_3 phase in the gas mixtures of the present studies. Similarly, reaction between Al_2O_3 and Cl_2 or HCl would result in negligible volatile AlCl_3 phase at 650°C . Reaction between Cr_2O_3 and Cl_2 would lead to significant pressure of the volatile CrCl_3 phase, but reaction between Cr_2O_3 and HCl is not favored in the gas environments of the present study. The reaction between NiO and Cl_2 or HCl would result in the solid NiCl_2 phase with a vapor pressure of 5×10^{-4} atm at 650°C . The calculations indicate that for the alloys to perform with good corrosion resistance, direct reaction between Fe, Cr, and Al with Cl_2 or HCl must be minimized. Further, even among the oxides, Cr_2O_3 seems susceptible to attack in Cl_2 but oxides of Fe and Al are beneficial in minimizing corrosion. Reactions between Ni and/or NiO and Cl_2 or HCl would form the solid NiCl_2 phase, the primary cause for Ni-containing alloys to perform better in HCl -containing environments that was observed in the ANL study.

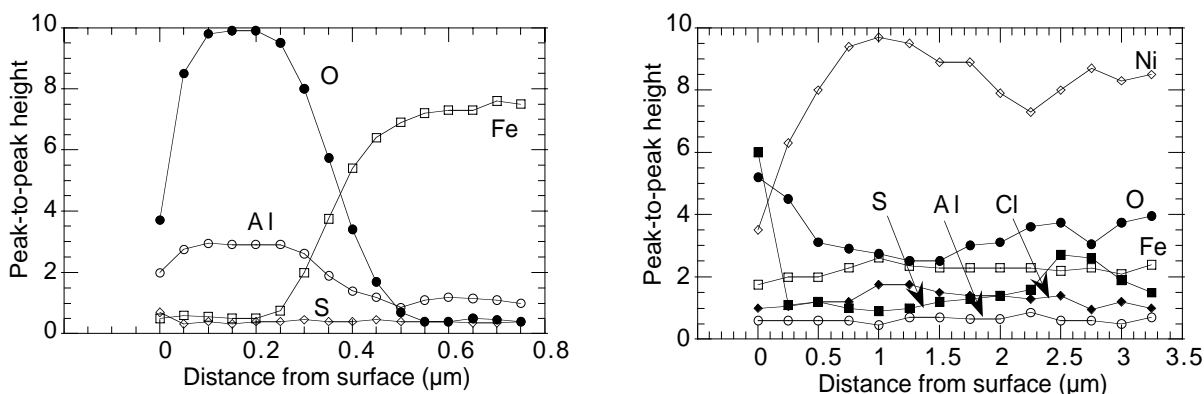
Performance of Iron Aluminides

Iron aluminide intermetallics are being developed for use as structural materials and/or as cladding for conventional engineering alloys. In addition to their strength advantages, these materials exhibit excellent resistance to corrosion in single-oxidant and perhaps in multioxidant environments at elevated temperatures through the formation of slow-growing, adherent alumina scales. Even though these intermetallics develop protective oxide scales in single-oxidant environments, the simultaneous presence of several reactants in the environment can lead to development of oxide scales that are nonprotective and that undergo breakaway corrosion, or to nonoxide scales that are detrimental to the performance of the underlying alloy. The oxidation resistance of iron aluminides depends on the formation of a chemically stable Al_2O_3 surface layer upon exposure to an oxidizing environment. The Al levels present in Fe aluminides (15.9 and 20-30 wt.% in Fe_3Al and FeAl , respectively) are well in excess of the critical concentration needed for the formation of a continuous alumina scale on the surface. However, at lower temperatures, transient Fe oxides will be present and the thermodynamically stable alumina can develop a continuous scale only over long exposure times. Extensive research has been conducted to evaluate the oxidation/corrosion performance of bulk Fe aluminide alloys and overlay coatings of Fe aluminides in single- and multioxidant environments.

Two distinct modes of failure are identified in the breakdown of alumina scales. The first is due to transport of iron outward from the substrate, through the scale, to the gas/oxide interface, a sequence of events that leads to the formation of Fe oxide nodules on the scale surface. These nodules, which eventually coalesce and cover the entire surface, form irrespective of whether the scale is α -alumina or some other low-temperature alumina. Our study indicates that from the standpoint of additions of elements such as Zr, Nb, etc., the composition of the Fe aluminide has a negligible effect on Fe transport. Another concern at low temperatures is that the alumina scaling rate may be so low that transient oxides of Fe, Cr, and other elements may be present in the corrosion-product scale for a long period of exposure and thus the benefit of slow-growing alumina for corrosion protection may not be realized in practice. The second mode of failure is due to mechanical separation of the scale from the substrate. The mechanical stability or the lack of it, a well-known problem associated with alumina scales, is also a problem for Fe aluminides. Scales composed of alumina commonly exhibit mechanical instability, e.g., cracking, spallation, and delamination, which poses a serious drawback in the application of Al_2O_3 -forming alloys.

Extensive studies have been conducted on the corrosion performance of alumina-forming alloys in O/S mixed-gas environments. The results showed that a critical Al content in excess of 12 wt.%, which is present in Fe aluminides, is needed for the formation of alumina on the alloy surface in environments that are typical of coal conversion systems. The studies included evaluation of these materials in complex environments that simulate coal gasification (characterized by low $p\text{O}_2$ and S present as H_2S) and coal combustion (characterized by high $p\text{O}_2$ and S present as SO_2). All of the Fe aluminide coatings were resistant to sulfidation and chloride attack, whereas the base alloys were susceptible to general corrosion and pitting attack, especially in the HCl-containing environment. Even in the combustion atmospheres, the Fe aluminides showed a small weight gain due to the development of a thin, adherent alumina scale; however, in the presence of HCl, the alloys/coatings of Fe aluminide exhibited substantial attack. To examine the cause(s) for the increased corrosion of the Fe aluminide in

HCl-containing environments, depth profiles for several of the pertinent elements in the scale were obtained by Auger analysis after sputtering the surfaces for various times. The next figure shows peak-to-peak height as a function of depth for Fe₃Al-coated specimens after exposure in air/SO₂ and air/SO₂/HCl environments at 650°C. When Cl is present in the exposure environment, several constituents of the coating and substrate alloy can react to form volatile chlorides, an occurrence that can lead to loss of the oxide-forming elements. The results showed that the driving force for the reaction between Fe and Cl₂ or HCl is sufficiently high in the simulated combustion environment to form volatile FeCl₃. Similarly, the driving force for the reaction between Al and Cl₂ or HCl is sufficiently high in the simulated combustion environment to form volatile AlCl₃, Al₂Cl₆, and AlOCl phases. The results also indicate that to minimize the formation and escape of volatile chlorides of oxide-forming elements, the alloys must form the stable oxides early in the oxidation process.



Auger peak-to-peak height data for several elements as a function of depth for scales developed on Fe₃Al coating on Type 316 stainless steel after exposure to (left) air/SO₂ and (right) air/SO₂/HCl environments at 650°C

Oxidation Behavior of Molybdenum Silicides

High-temperature structural materials are critically needed for the improvement of the thermal efficiency and reliability of energy conversion systems and advanced engine systems. Currently available alloys, such as Ni-based single-crystal superalloys, are limited to use at temperatures of ≈1100°C. Superalloys derive their intrinsic strength from reinforcements of gamma prime precipitates, but they tend to coarsen and ultimately dissolve as the temperature increases beyond 1100°C. NiAl-based aluminide alloys, which are currently under development, have potential for use at temperatures up to 1200°C. However, many applications require temperature capabilities that exceed this temperature by at least 200°C. The melting temperature (T_M) of a material for structural applications at 1400°C should be >2000°C so that at most, 0.75 T_M is reached during service and appreciable high-temperature strength is maintained. Of the potential candidate systems, Mo silicides are particularly attractive because of their high melting points. Molybdenum disilicide exhibits particularly good mechanical strength properties, high thermal conductivity, high electrical conductivity, and promising oxidation resistance at elevated temperature. However, the material has been reported to exhibit high creep rates at temperatures of >1200°C and degradation by a phenomenon known as “pesting” in the temperature range of 400-600°C.

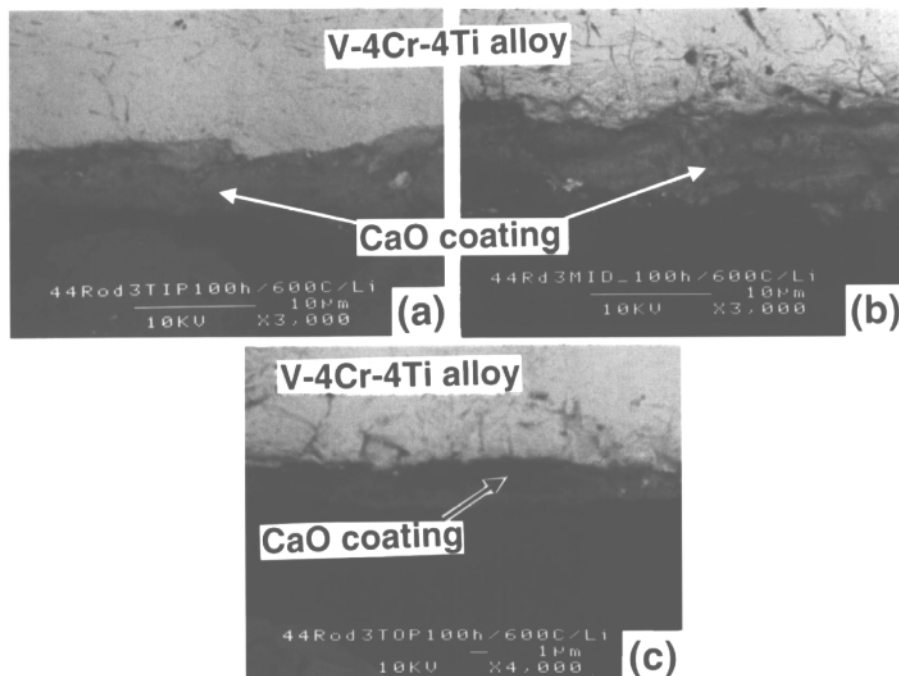
Oxidation tests were conducted on Mo-based silicides over a wide temperature range to evaluate the effects of alloy composition and temperature on the protective scaling characteristics and peeling regime for the materials. The study included Mo_5Si_3 alloys that contained several concentrations of B. In addition, oxidation characteristics of $\text{MoSi}_2\text{-Si}_3\text{N}_4$ composites that contained 20-80 vol.% Si_3N_4 were evaluated at 500-1400°C. Results showed that binary Mo silicides are prone to nonprotective oxidation at low temperatures (500-700°C) in air, primarily because silica growth rates to form an external continuous scale are extremely low. Additions of more stable oxide-forming elements may only have a marginal benefit at low temperatures because the growth rates of most of those oxides are also low. Addition of B to accelerate formation of borosilicate at low temperatures is not proved. Silica (or borosilicate) scale seems to peel at elevated temperatures (1000-1400°C), indicating significant plasticity; however, long-term protective capacity of the scale may be degraded. The oxidation behavior of silicide-ceramic composites (e.g., $\text{MoSi}_2\text{-Si}_3\text{N}_4$) is excellent in the temperature range of 800-1400°C and the rates for the composites are significantly lower than those observed for B-containing Mo_5Si_3 alloys. Even the composites exhibit peeling or nonprotective scaling at lower temperature (e.g., 500°C), suggesting that the ceramic component is inert from the oxidation standpoint and that the oxidation resistance of the composite is determined predominantly by that of the silicide in the composite.

Fusion Energy

Vanadium alloys have been identified as leading candidate materials for fusion first-wall/blanket structure applications. Current research is directed toward development of electrically insulating coatings for the V alloy, development of uniaxial creep data on several heats of V alloys, thermodynamics and kinetics of oxygen and hydrogen ingress into V alloys and their effect on mechanical properties, chemical compatibility of structural alloys and coatings in a Li environment, and development of design criteria for advanced fusion concepts.

Electrically Insulating Coatings

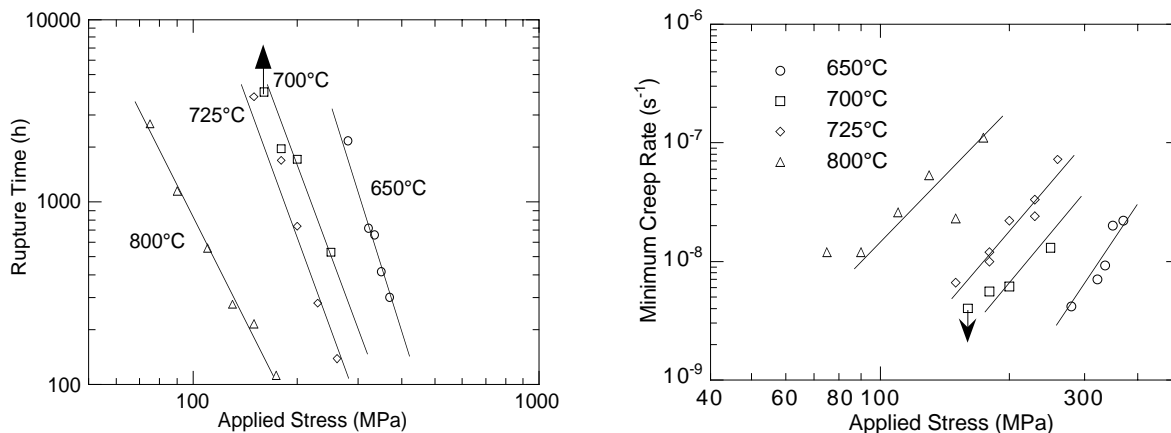
The main challenge in the design of self-cooled blankets is accommodation of the strong influence of the magnetic field on the liquid-metal flow. Based on the thermodynamics of interactions between the coating and the liquid lithium on one side and the structural V-base alloy on the other side, several coating candidates are being examined to perform the insulating function over a wide range of temperatures and lithium chemistries. We have developed CaO coatings by a thermal/chemical vapor deposition process and by an in-situ approach in a liquid Li-Ca environment. Results showed that thick adherent coatings can be fabricated by thermal/chemical vapor deposition, especially if a double Ca treatment is applied. Extensive microstructural analysis of the coatings developed by the thermal/chemical process showed almost 100% CaO over a coating thickness of 20-30 μm and the electrical resistance, measured by two-probe method, of these coatings was at least two orders of magnitude higher than the minimum required for blanket application. The next figure shows SEM photomicrographs of various regions of a CaO-coated rod specimen after 100-h exposure to an Li-10 at.% Ca environment.



SEM-photomicrograph cross sections of (a) tip, (b) middle, and (c) top portions of 70-mm-long V-4Cr-4Ti rod specimen after exposure to Li-10 at.% Ca at 600°C for ≈100h. Specimen was coated with CaO by double Ca deposition/oxidation in He flow

Uniaxial Creep Properties of V-Base Alloys

Refractory alloys based on V-Cr-Ti are being considered for use in first-wall structures in advanced blanket concepts that use liquid Li as a coolant and breeding material. The long-term creep properties of the V-base alloys will be influenced by the time-dependent nucleation and growth of precipitates that involve nonmetallic elements such as O, N, and C. Several microstructural studies of V-base alloys have identified precipitates such as face-centered-cubic Ti(O, N, C) with variable O, N, and C ratios. To correlate microstructural development with creep properties, we plan to establish the time-dependent evolution of type, number, and location of precipitates in V-base alloys. Furthermore, development of several of these precipitates can be influenced by the exposure environment during creep testing. Over the long term, creep data will be obtained in environments with a wide range of chemistry and that encompass high vacuum to low partial pressures of O and H, and He of various purities. A systematic study is currently being conducted at ANL to evaluate the uniaxial creep behavior of V-Cr-Ti alloys as a function of temperature in the range of 650-800°C and at applied stress levels in the range of 75-380 MPa, with emphasis on baseline creep behavior of the alloys and correlations between microstructures and properties. The next figure shows typical results obtained on time to rupture and creep rate as a function of applied stress at several temperatures.

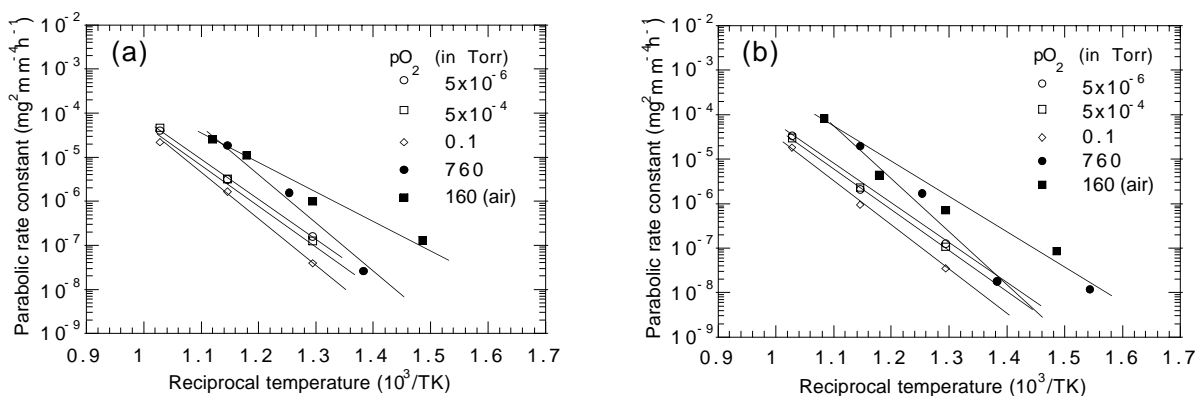


Variation of rupture time (left) and minimum creep rate (right) as a function of applied stress for V-4Cr-4Ti alloy creep tested at 650-800°C

Oxidation Performance of V-Base Alloys

A primary deterrent to the use of V-Cr-Ti alloys at elevated temperatures is their relatively high affinity for interstitial impurities, i.e., O, N, H, and C. We conducted a systematic study to determine the effects of time, temperature, and oxygen partial pressure (pO_2) in the exposure environment on O uptake, scaling kinetics, and scale microstructure in V-(4-5) wt.% Cr-(4-5) wt.% Ti alloys. Oxidation experiments were conducted on the alloys in a pO_2 range of 5×10^{-6} -760 torr (6.6×10^{-4} - 1×10^5 Pa) at several temperatures from 350 to 700°C.

Models that describe the oxidation kinetics, oxide type and thickness, alloy grain size, and depth of O diffusion in the substrate of the two alloys were determined and compared. Weight change data were correlated with time by a parabolic relationship. The parabolic rate constant was calculated for various exposure conditions, and the temperature dependence of the constant was described by an Arrhenius relationship (see next figure). The results showed that the activation energy for the oxidation process is fairly constant at pO_2 levels in the range of 5×10^{-6} -0.1 torr. The activation energy calculated from data obtained in the air tests was



Temperature dependence of parabolic rate constant for O uptake of (a) V-4Cr-4Ti and (b) V-5Cr-5Ti alloys in several low- pO_2 and air environments.

significantly lower, whereas that obtained in pure-O tests (at 760 torr) was substantially higher than the energy obtained under low- pO_2 conditions. The oxide VO_2 was the predominant phase that formed in both alloys when exposed to pO_2 levels of 6.6×10^{-4} to 0.1 torr. V_2O_5 was the primary phase in specimens exposed to air and to pure O_2 at 760 torr. The implications of the increased O concentration are increased strength and decreased ductility of the alloy. However, alloy strength was not a strong function of the O concentration of the alloy, but an increase in O concentration did cause a substantial decrease in ductility.

Design Criteria and Materials Properties

ANL has had primary responsibility since 1995 for the development of new design rules, including those to handle irradiation embrittlement. During 1998, the final draft of the ISDC was issued. Following completion of the EDA phase of the International Thermonuclear Reactor, staff in this Section have been involved in extending the design rules to high-temperature applications for advanced blanket designs currently under study in the U.S.

Design criteria input has also been supplied to the U.S. ARIES Team regarding rules for materials such as copper alloys, which embrittle significantly at relatively low neutron damage levels. Materials data input for ARIES-ST has been provided for copper alloys, aluminum alloys, low-activation ferritic steels, silicon carbide, and tungsten.

Stress analysis and design criteria require well-developed data base for the candidate structural materials. The Advanced Materials Section of DOE's Office of Fusion Energy Sciences is actively supporting mechanical property research into reduced activation ferritic/martensitic steels, V alloys, and monolithic and composite SiC. The staff of this Section have the lead U.S. role as interface between the design communities and the materials community. This involves assessing the data bases and presenting them in a usable, relevant form to U.S. design teams. In addition, an active role is played in the assessment of the vanadium-alloy data base needs and the planning and post-test analysis of additional experimental work performed at ANL in the areas of tensile, impact, and creep properties.

Office of Industrial Technologies

Metal dusting is a catastrophic corrosion phenomenon that leads to the disintegration of structural metals and alloys into dust composed of fine particles of the metal/alloy and carbon. This phenomenon has been observed in the chemical and petrochemical industries, in reformer and direct-reduction plants, in processes that generate syngas, and in other processes where hydrocarbons or other high-carbon-activity atmospheres are present. Failures have been reported in ammonia plants as reduced energy requirements result in a lower steam/ H_2 ratio while CO/ CO_2 ratios have tended to increase. Even though metal dusting is widely prevalent, the general approach to minimize the problem in industry is to avoid the temperature/process conditions that are conducive for the attack, usually at a penalty in production, efficiency, and cost. Also, fixes such as sulfur poisoning of surface sites, preoxidation of alloy to stabilize chromia on high-Cr alloys, etc., are applied case-by-case, primarily based on experience with performance of materials in such environments. DOE's Office of Industrial Technologies is supporting a three-year project in cooperation with several industrial partners to address the metal dusting problem from a fundamental scientific base using laboratory research in

simulated process environments and subsequent field testing of materials in actual process environments with participation from the U.S. chemical industries.

The effort at ANL would involve laboratory testing of materials in simulated process environments, development of a user-friendly computer-based data base on metal dusting/corrosion/mechanical properties of materials, and development of a fundamental understanding of the metal dusting and associated carburization phenomena. Alloy manufacturers would contribute materials for both laboratory and field testing, participate in the conduct of corrosion tests at ANL in addition to their in-house evaluation of materials, and assist in microstructural evaluation of tested specimens. Air Products and Chemicals Inc. and DuPont Company and others would assist in establishing the test conditions for the laboratory program at ANL, provide information on actual plant operating conditions, provide suggestions on materials to test, and assist/coordinate actual field testing of materials. Tube sections of the best alloys tested and/or surface-engineered material with adequate metal dusting resistance will be evaluated by exposure in a commercial hydrogen plant operated by Air Products and Chemicals, Inc.

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Draft for Public Comment Prepared for NRC, Office of Nuclear Reactor Regulation, by
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Generic Aging Lessons Learned (GALL) Report

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Irradiation Performance

Section Manager: T. S. Bray

Principal Investigators: M. C. Billone, H. Tsai, R. V. Strain, Y. Yan, R. S. Daum

Salaried Staff and Technicians: L. F. Essenmacher, W. C. Kettman, D. J. McGann,

D. O. Pushis, D. P. McGann, A. D. Storey

Secretary: S. M. Baumann

The activities in the Irradiation Performance Section (IPS) are aimed at determining and assessing the behavior of neutron-irradiated materials throughout the life cycle of the materials from their in-reactor performance through their likely behavior during ultimate interment in a repository. This environment includes neutron damage and chemical, metallurgical, and mechanical processes that occur during operation in fission or fusion reactors. Subsequent environments could include long-term storage in either water or air, with degradation mechanisms attendant to these environments, together with the consequent concerns for maximum containment of the radioactive species. To accomplish these tasks, the Section operates and utilizes two major facilities: the Alpha-Gamma Hot Cell Facility (AGHCF), which includes the well-equipped Electron-Beam Instrument Laboratory (EBL); and the Irradiated Materials Laboratory (IML). Minor facilities are the glovebox facility in Room DL-114 and the K-2 Cell in Building 200 M-Wing. Characterization and testing of irradiated materials is carried out in the AGHCF and the IML. We have refurbished the DL-114 glovebox facility to carry out hydrogen, nitrogen, and oxygen measurements for our programs. Finally, in early 2001, we reached an agreement with another ANL Division to take over a portion of the Building 200 M-Wing Hot Cell Facility to allow for multiple and simultaneous mechanical properties tests to be conducted.

Fission Reactor Materials

The Section's activities in the fission reactor area are focused principally on light water reactors (LWRs) but also include work on test reactor fuels and materials and advanced naval reactor materials. The capabilities of the Section's facilities are well suited for research in these areas. The principal ongoing and upcoming activities in fission-reactor research are described below.

High-Burnup Cladding Performance

The NRC/RES-sponsored program, "High-Burnup Cladding Performance" is a major long-range research effort in the Section with participation also by staff from ET's Corrosion Section. The purposes of this program are to determine the behavior of LWR fuel rods under conditions relevant to Loss-of-Coolant Accidents (LOCA) and to establish a mechanical properties data base for high-burnup (>60 MWd/kg) Zircaloy cladding. These data are needed for the analysis of various transients, including LOCA, Reactivity-Initiated-Accidents (RIA), and Anticipated Transient Without Scram (ATWS), and are important in licensing-safety analyses. To accomplish these objectives, the project is divided into three major tasks: Fuel Selection and Characterization, LOCA-Related Tests, and Mechanical Properties Tests.

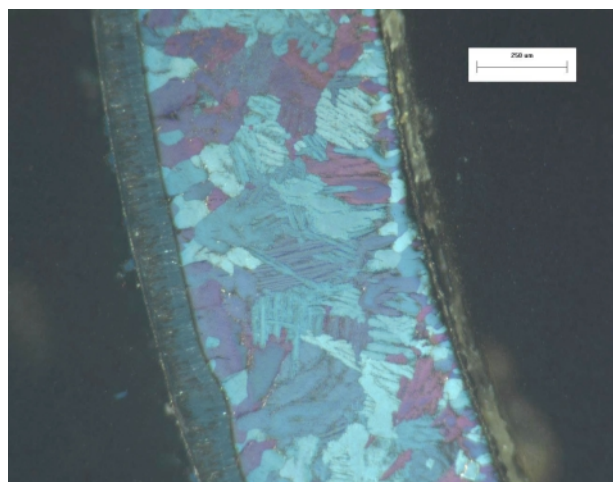
Fuel Selection and Characterization

Under a special agreement between the Electric Power Research Institute (EPRI) and NRC/RES, EPRI has agreed to supply segments of seven high-burnup Zircaloy-2-clad boiling water reactor (BWR) fuel rods and segments of seven high-burnup Zircaloy-4-clad pressurized water reactor (PWR) fuel rods. Both fuel and cladding will be characterized by the IPS. Working directly with NRC/RES and EPRI, the Section has been actively involved in the rod selection process. The BWR rod segments were received at ANL in May 2000. Earlier, PWR fuel rod segments (≈ 50 MWd/kg) from the TMI-1 reactor were used to demonstrate our ability to characterize fuel and cladding and to validate the experimental procedures for characterization, as well as to conduct all aspects of the program. Data from the TMI-1 examinations are also useful for increasing our understanding of fuel and cladding behavior at intermediate burnup.

LOCA-Related Tests

This task involves high-temperature oxidation experimental and analytical studies, LOCA-criteria tests, and structural response tests. Progress has been made in designing and validating the high-temperature (900 - 1300°C) oxidation equipment and experimental procedures, as well as demonstrating this capability in the AGHCF by testing the TMI-1 rod cladding (see figure below). Steam oxidation tests have been performed on unirradiated and irradiated BWR cladding samples at the LOCA-criteria temperature of 1204°C .

The LOCA-criteria tests involve ramping (at a rate of $\approx 15^{\circ}\text{C/s}$) the temperature of fueled cladding segments in a flowing-steam environment to 1204°C , holding for various times to generate oxidation levels consistent with NRC criteria (17% Equivalent Cladding Reacted), slowly cooling (at a rate of $\approx 15^{\circ}\text{C/s}$) to $\approx 700^{\circ}\text{C}$, and rapid cooling through water-quenching. Behavior of the cladding during the water-quench determines whether the current NRC criteria for LOCA events are conservative for high-burnup fuel rods. Similar tests were performed at ANL more than 20 years ago on unirradiated cladding, but the challenge of the current program is to design equipment and procedures suitable for remote operation in the AGHCF. Investigators from the IPS work closely in this area with staff from the Corrosion Section. This major subtask is now in the detailed engineering design stage following extensive out-of-cell experimental work with a mock-up apparatus. The third subtask within this area requires slow and rapid bending of high-burnup-fueled cladding segments to determine mechanical properties and failure thresholds for analysis of seismic and LOCA-blowdown events, as well as post-LOCA seismic events. Recently, a fourth subtask was added to determine post-LOCA ductility by using ring-compression and/or tensile samples.

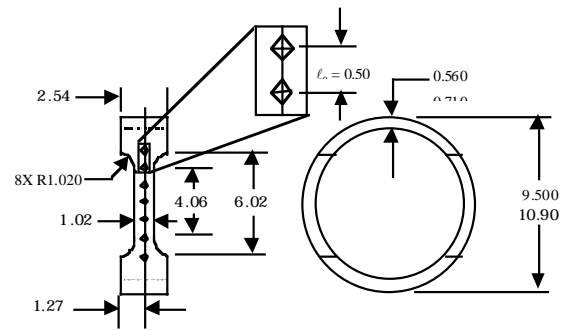


As-polished surface of irradiated Zircaloy cladding after exposure to steam at 1204°C for 10 minutes

Mechanical Properties Tests

Tensile tests with high and low strain rates (for RIA and LOCA, respectively) are planned to determine the mechanical properties of BWR and PWR Zircaloy cladding in both the hoop and axial directions (see next figure). In addition, biaxial tube burst tests are planned for fueled cladding at high strain rates (RIA) at several hoop:axial stress ratios. Collaboration with researchers at Pennsylvania State University has been established in the area of tensile testing.

Extensive finite-element analyses have been conducted by S. Majumdar of the Corrosion and Mechanics of Materials Section to determine the optimal specimen geometry for both uniaxial and plane-strain tensile tests. Because the hoop tensile tests are extremely difficult to conduct and their results are difficult to interpret, the French, Russian, and Japanese experiences in this area have been reviewed in detail as part of the test planning. The primary goal of this effort is to supply fuel performance modelers with much-needed data for their computer codes, and therefore a special subtask has been established for the interface between the data base and the modeling. An extensive review of the current data base has been conducted to determine its status, the modeling needs beyond the current data base, and anticipated additions to the data base from the IPS mechanical properties tests. The TMI-1 PWR cladding has been used to demonstrate IPS capabilities of performing hoop tensile tests with irradiated cladding ring samples.



Ring tensile specimen configuration

Dry Cask Storage

The IPS is investigating spent-fuel behavior during dry cask storage. Certain issues remain unresolved for criteria on 20-year dry-cask storage of high-burnup fuel, as well as for the extension of storage times to >20 years for intermediate-burnup fuel. These issues relate to effects of high-burnup degradation on cladding integrity during 20-year storage and the effects of longer storage times for both intermediate- and high-burnup cladding. Improvements in the mechanical properties data base, particularly for thermal creep, and in modeling are needed to provide a technical basis for dry cask storage criteria.

For intermediate-burnup fuel rods (≈ 45 MWd/kg), IPS has an active program in characterizing, conducting axial tensile tests, and long-term thermal creep tests of PWR cladding (from the Surry Reactor) that has been irradiated to 36 MWd/kg and stored in a helium-filled cask for 15 years. Long-term thermal creep tests will be conducted to determine the residual creep strain-to-failure, creep strain vs. time, and secondary creep rate, in a test apparatus shown in the next figure.

For the high-burnup portion of this work, two additional H. B. Robinson PWR rods (70 MWd/kg) will be used for sample selection, preparation, and testing. The additional rods will be procured under the baseline program through the agreement between NRC and EPRI. Sample characterization will use fuel isotopic analysis, metallography, measurement of oxide layer thickness, and hydrogen morphology. Thermal creep tests will use the same procedures

developed for the intermediate-burnup testing program, but with a different focus. Because the central licensing issue for high-burnup rods is the first 20 years of dry cask storage, the generation of baseline thermal creep data vs. stress, temperature, time, and hydrogen content is more important than determination of residual creep life. Isotopic composition of high-burnup fuel is important in determining the neutron multiplication factor for criticality analyses, as well as decay heat. Code modelers at Oak Ridge National Laboratory have identified the isotopes of interest for both BWR and PWR fuel analyses. ANL's Chemical Technology Division will conduct the work with funding provided by either the Yucca Mountain Program or the NRC.

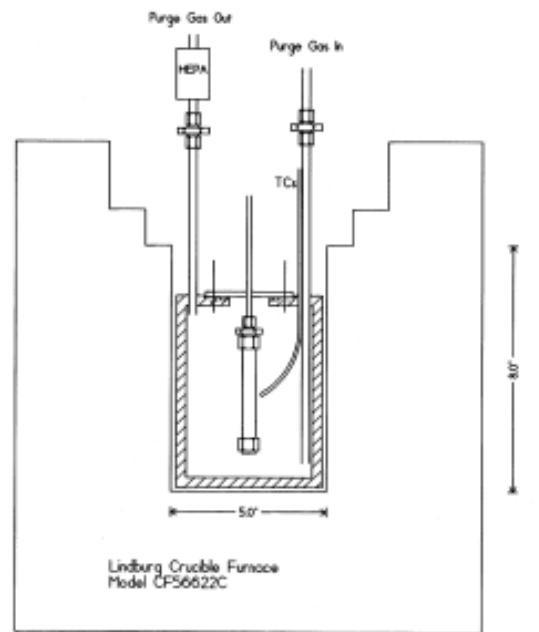
Zinc/Zircaloy Interaction

Due to limited capacity in spent-fuel pools, some nuclear power plants are storing spent-fuel assemblies in inert-atmosphere dry storage casks, such as the VSC-24 cask. Because the casks are submerged in the spent-fuel pool water during the spent-fuel loading process, a zinc-based primer coating is sometimes applied to the exposed cask structure to protect the steel from rust and corrosion. The relatively high vapor pressure of zinc at the cask handling and storage temperatures, however, may cause a metallurgical interaction with the fuel rod's Zircaloy cladding, potentially affecting cladding performance in storage. The objective of this project is to determine the extent of such interaction by conducting tests simulating cask handling and storage conditions.

Aluminum-Clad Spent Nuclear Fuel

The long-term storage and ultimate disposition of aluminum-clad fuel from production reactors and domestic and foreign test reactors is the responsibility of DOE's Savannah River Site (SRS). Aluminum-clad fuels include U-Al alloys and UAl_x , U_3O_8 , and U_3Si_2 dispersions in an aluminum matrix. Since 1996, we have assisted SRS in defining acceptance criteria for fuel that will be put into long-term storage at SRS and in assessing the potential problems that may develop during either storage or disposal. The issues are the current corroded state of the cladding after years in wet storage, an acceptable cladding corrosion rate in the dry environment of the long-term storage, possible fission-product-driven fuel degradation during storage, fission-product release rates from degraded fuel, and fission-product release during possible high-temperature treatment.

One possible method of high-temperature treatment is melt-dilution, in which the spent Al-clad fuel is melted (with or without additives) to dilute the enriched uranium. Melt-dilution is attractive because it reduces proliferation and criticality concerns and may enhance corrosion resistance of the material for storage in a repository. The main issue with this treatment method is the release of volatile fission products, e.g., cesium, during the melting process.



Thermal creep test chamber and specimen conceptual design

Effective entrapment of the released fission products is essential. SRS is developing a pilot-scale melt-dilution facility in which whole assemblies will be treated.

To support the development of the melt-dilution process, the Section conducted three radiant-furnace tests with irradiated UAl_x fuel at 850°C in the AGHCF. The getter materials to trap the released volatile fission products were activated aluminate in one test and Zeolite in the two others. The results showed that Zeolite can be an effective trapping media. These results are highly encouraging. We did encounter one difficulty in our tests, namely, the oxide layer on the surfaces of the molten fuel/Al ingots prevented proper mixing of the ingots. We have assembled an induction-furnace-based test apparatus to increase mixing and the melt size. Two tests are planned in 2001 for whole-miniplate test specimens, a scale-up of approximately 30%.

Reduced Enrichment Research and Test Reactor Fuel

The goal of the Reduced Enrichment Research and Test Reactor (RERTR) program is to develop low enrichment uranium (LEU, i.e., <20% ^{235}U enrichment) fuels to replace the high enrichment uranium (HEU) fuels commonly used in research and test reactors around the world. A high-density U_3Si_2 dispersion fuel, with a uranium loading density of up to 4.8 g/mL in an aluminum matrix, has been developed by ANL and approved by NRC. This loading density is sufficient to convert nearly 90% of the research reactors that use HEU of U.S. origin, and many research reactors have already been successfully converted to LEU. For some research and test reactors, however, the loading density of the U_3Si_2 fuel is not sufficient. In order not to derate these reactors, an advanced LEU fuel with a uranium loading density of 8-9 g/mL is necessary. This requires a very dense fuel dispersant (>15 g U/mL) and a very high volume loading of the dispersant (>50 vol.%). The Section has one important role in the RERTR program: to examine and evaluate the fuel plates after the irradiation.

We have completed the nondestructive and destructive examination of 64 irradiated microplates from RERTR-1 and RERTR-2. The nondestructive examinations included visual inspection, thickness measurements, weight measurements, and gamma scanning. Optical microscopy, scanning electron microscopy (SEM), electron microprobe analysis, and blister tests were used for destructive examination. Results from optical microscopy and SEM have shown that the uranium-molybdenum alloys (between 6 and 10 wt.%) were well behaved at burnups of about 40 and 70%. That is, these alloys show low interaction with the aluminum matrix and low fission-gas swelling manifested in a uniform distribution of very small fission gas bubbles. In contrast, the U-5%Nb-3%Zr fuel exhibited extensive interaction with the matrix and unstable fission-gas-bubble behavior.

A third irradiation experiment, RERTR-3, was completed to test the more promising fuel designs from the RERTR-1 and -2 experiments at higher temperatures and higher fuel loadings. New matrix materials with the potential to further reduce fuel/matrix interactions were also tested in the RERTR-3 experiment. A promising candidate matrix material is magnesium and a method of producing fuel plates with a magnesium matrix with aluminum cladding has already been demonstrated by the IPS. Postirradiation examination of the RERTR-3 nanoplates is being conducted in the AGHCF. Examination has included optical microscopy and SEM. Results indicate that bubble formation on the grain boundaries of fuel receiving an 800°C heat treatment was greatly reduced when compared to fuel receiving a 500°C heat treatment or no heat treatment. Annealing at 500°C results in the formation of a fine

microstructure composed of intermediate and equilibrium decomposition products. This allows for easier fission-gas-bubble nucleation. The uniform microstructure in the homogenization-annealed specimens is apparently a significant barrier to fission-gas-bubble nucleation. The interdiffusion of the U-Mo alloy fuel and the matrix aluminum is being characterized. In the highest-temperature samples of RERTR-3, substantial volume fractions of interaction product were observed with a concomitant depletion of the matrix aluminum. This interaction is, at high temperature, the major contributor to fuel plate swelling and degrades the fuel meat thermal conductivity. This increases fuel meat temperature and thus enhances interdiffusion. Detailed measurements of the extent of interaction were made over the entire fuel temperature range tested in RERTR-1, -2, and -3.

Two irradiation experiments, RERTR-4 and RERTR-5, are underway. RERTR-4 was discharged from the reactor in January 2001 and will be received for postirradiation examination at the AGHCF in early March 2001. RERTR-5 will continue irradiation to its target burnup of 80%, to be reached in late summer of 2001.

Fusion Reactors

Advanced Fusion Structural Materials

The IPS continues to play a lead role in ANL's fusion reactor structural materials program. The objective of the program is to develop low-activation vanadium alloys for the fusion reactor's first-wall and blanket structures. We have continued the testing of specimens from a variety of origins. Most recently we have determined laser weld parameters for welding ≈ 4 -mm-thick plate of V-4Cr-4Ti alloys, prepared laser weld specimens for mechanical testing, measured tensile and impact properties of V-(Cr,Fe)-Ti alloys, evaluated the effect of heat treatment on the microstructure of V-4Cr-4Ti alloys, completed quantitative oxygen analyses for V-4Cr-4Ti alloys, and determined the impact properties of V-4Cr-4Ti laser weldments.

Results from the above work include determining via metallography that grain sizes in the welded specimens tend to be greater than those in the base materials. Impact testing of these specimens showed that a ductile-to-brittle transition temperature for both the base metal and the laser weld is $\approx -20^\circ\text{C}$. Results from postirradiation tensile tests on V-(Cr,Fe)-Ti alloy specimens irradiated in the HFIR-11J experiment at $\approx 300^\circ\text{C}$ show significant radiation hardening and ductility reduction in all of the materials. In comparison, hardening was substantially lower in the same materials after irradiation in the 12J experiment, which was conducted at 500°C . ANL Heat 832665 tensile and Charpy specimens exposed in the DIII-D experimental tokamak were tested; results indicate no significant degradation of the mechanical properties of this alloy.

Work will continue in this area with particular attention given to determining properties of these advanced materials. The Section is also contributing to the design and implementation of the new JUPITER II irradiation experiment. This work design and fabrication of specimens is planned for 2001 with insertion into the ATR or HFIR reactor in 2002.

Other Current Activities

The Section is actively working in a number of other research areas. We are beginning the multiyear Nuclear Engineering Plant Optimization program that will include design and construction of a constant extension rate testing system. This new system will be tested on unirradiated stainless steel materials; irradiated specimens will eventually be fabricated by using our Section's electric discharge machine (EDM) capability. We are also working on a NERI program in which we have revitalized our Auger microprobe to allow for fracture surface examination of proton-irradiated stainless steel specimens; the goal is to determine the composition of grain boundary surfaces relative to that of the bulk material, as well as effects of composition on mechanical properties. We are also continuing the tensile testing of irradiated stainless steel under our CRIEPI programs. Those specimens are also fabricated by using our EDM capability.

Ancillary Activities

Component Failure Analysis

The IPS performs failure analysis on radioactively contaminated components, or pieces, removed from service in reactors operated by Commonwealth Edison Co. (ComEd). These analyses, which consist only of direct metallurgical examinations and tests, support ComEd's in-house analytical efforts to determine the causes of component failures. The Section provides ComEd with quick response over a range of services, unavailable elsewhere in the area, for radioactive materials.

Examination Support

The Section is continuing its collaboration with the Corrosion Section in the NRC's Irradiation-Assisted Stress Corrosion Cracking Program for LWR stainless steel components. Our efforts provide the investigators with the necessary fractographic (SEM) and microchemical (Auger microprobe) data for evaluating the phenomenon.

Future Directions

The strength of the Section continues to be our expertise in irradiation experiments and characterization of irradiated fuels and materials. While maintaining our competency in fuels-related research, we are expanding our capabilities into the area of mechanical testing of irradiated materials. Recently established programs such as the NRC LOCA and related testing programs, the ANL/JNC/CRIEPI stainless steel program, and the fusion materials testing program, fit into this general direction. We have also submitted numerous proposals in the NERI and NEPO programs. To handle the increased space requirements, we have obtained additional hot cell space for mechanical testing in Building 200. It is recognized, however, that because of the dwindling nuclear market and the aging of our facilities, there are significant challenges ahead. Maintaining the Section's core competency in working with radioactive materials for both DOE and NRC materials programs is our goal. Sharing these resources and capabilities with industry to complement their own facilities and needs is a recognized path forward. Finally, to ensure that our capabilities are maintained, we are focused on improving and transitioning our human resources; we have obtained three new technicians and two new

staff in the past two years. In addition, we are actively searching for two additional staff members. The acquisition of the appropriate employees will ensure that we have the resources available to maintain our capabilities.

Recent Work by the Irradiation Performance Section

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M. A. Kirk and Y. Yan

Micron 30 (1999) 507-526.

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A. S. Wagh, R. Strain, S. Y. Jeong, D. Reed, T. Krause, and D. Singh

Journal of Nuclear Materials 265 (1999) 295-307.

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Y. Yan and M. K. Kirk

Phil. Mag. Lett. 79(10), 841-848 (1999).

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J. Am. Ceram. Soc. 83 (5) 1266-72 (2000).

Irradiated UAl_x Melt Dilution Testing at Argonne National Laboratory

T. S. Bray, A. B. Cohen, R. V. Strain, H. Tsai, and T. Adam (SRL)

Presented at Minerals, Metals, and Materials Society 2000 Fall Mtg., St. Louis, MO, Oct. 8-12, 2000.

Characterization of N-Reactor Fuel Element SFEC5.4378 at Argonne National Laboratory

T. S. Bray, M. Goldberg, R. V. Strain, H. Tsai, and J. Y. Park

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Tensile and Impact Properties of V-4Cr-4Ti Alloy Heats 832665 and 832864

T. S. Bray, H. Tsai, L. J. Nowicki, M. C. Billone, D. L. Smith; W. R. Johnson, and

P. W. Trester (General Atomics)

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Presented at Waste Minimization Pollution Prevention Conf., Albuquerque, Nov. 15-19, 1999.

Stress Corrosion Cracking of Type 304 Stainless Steel Irradiated to Very High Dose

H. M. Chung, W. E. Ruther, R. V. Strain, and W. J. Shack

Paper No. 204, Corrosion 2000, Orlando, March 26-31, 2000.

Cracking Mechanism of Type 304L Stainless Steel Core Shroud Welds

H. M. Chung, J.-H. Park, J. E. Sanecki, N. J. Zaluzec; M. S. Yu, and T. T. Yang (Inst. for Nucl. Energy Res.)

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Irradiation-Assisted Stress Corrosion Cracking of Model Austenitic Stainless Steels

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T. S. Bray, H. Tsai, L. Nowicki, M. C. Billone, and D. L. Smith

Oral presentation at 4th IEA Vanadium Workshop, Argonne National Laboratory-East, April 21-23, 1999.

Tensile and Impact Properties of V-4Cr-4Ti Alloy Heats 832665 and 832864

T. S. Bray, H. Tsai, L. J. Nowicki, M. C. Billone, D. L. Smith, W. R. Johnson, and P. W. Trester

Oral presentation at 4th IEA Vanadium Workshop, Argonne National Laboratory-East, April 21-23, 1999.

Swift Heavy Ion Irradiation Damage in $\text{YBa}_2\text{Cu}_3\text{O}_{7-w}$ Superconductors

Y. Yan, R. Wheeler and M. A. Kirk

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Fractography Results for V-Alloy Tensile Specimens Irradiated in the BOR-60, ATR, and FFTF Reactors

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Study of Irradiation Creep of Vanadium Alloys

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Study of Irradiation Creep of Vanadium Alloys

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Tribology

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The Tribology Section conducts research on advanced tribological systems (e.g., surface engineered materials, lubricants, fuels, and fuel/lubricant additives) for use in aggressive environments. A major portion of this activity focuses on the development and evaluation of high-performance coatings that can be applied to a wide range of materials and how fuel and lubricant additives interact with surfaces under boundary-layer-lubrication regimes. The coatings are primarily intended to protect engine-component surfaces that undergo sliding and rolling contact in advanced transportation systems, including those powered by diesel and gasoline engines, as well as by advanced energy conversion systems being developed under the sponsorship of DOE's Office of Transportation Technologies and by U.S. industry. The overall goal of the Tribology Section is to promote industrial competitiveness by developing new tribological technologies and by solving problems associated with friction, wear, and lubrication. The Section has established and maintained close contact with transportation-related industries to determine their critical tribological needs and to facilitate the transfer of technologies developed at ANL.

The Section provides technical oversight for approximately \$6 million in programs for DOE's Office of Heavy Vehicle Technologies (OHVT). These programs include computational fluid dynamics modeling of tractor-trailers using a variety of eddy-drag models. Results are validated by NASA wind-tunnel experiments on a model tractor-trailer combination. The aerodynamic drag program also includes the development and testing of circulation control that will lead to a full-scale demonstration within 2 years. Wind-tunnel tests have demonstrated a 50% reduction in drag coefficient at 70 mph. Other major programs include friction and wear of engine components and brake materials, and thermal management, through use of nanofluids, evaporative cooling, and nucleated boiling. Technical oversight is also provided on three major new OHVT programs: "The more electric truck," an energy-efficient drive axle, and development of ceramic rotors and disks for disk brakes in heavy vehicles. The lead organizations on the winning competitive bids for the three new programs were Caterpillar, Dana, and Honeywell, respectively.

The Section's research activities include efforts on the development of surface modification processes, and evaluation of tribological properties. A significant effort focuses on the development of near-frictionless carbon (NFC) coatings and assessment of their friction and wear properties when used with engine components (in particular fuel injection systems) for advanced internal combustion engines and fuel cell compressor/expanders being developed for

the PNGV (Partnership for a New Generation of Vehicles) program. Other programs focus on the evaluation of solid lubricants for metal-forming operations, top-of-rail lubricants to minimize fuel consumption in railroad applications, lubricity additives for low-sulfur diesel fuels, assessment of boron-based compounds as low-friction fillers for polymers, development of NFC coatings for micro-electromechanical (MEM) devices, assessment of friction and wear properties of nanocrystalline diamond coatings, and characterization of aluminum casting dies. New efforts have been initiated to study the mechanisms by which fuel and lubricant additives interact with surfaces to form protective boundary layer films, and to evaluate the potential of laser glazing technologies for railroad applications. Numerous efforts are also underway with industry to develop NFC coatings for specific applications.

The following sections describe in greater detail the Tribology Section's research strengths and the programs that support activities in these areas. The major strength of the Section lies in its ability to not only develop processes to form novel coatings that are lubricious and wear-resistant, but more important, to perform detailed evaluation of the tribological properties of materials and coatings under a wide range of conditions that simulate those found in various energy-intensive sectors. This combined expertise in surface modification and tribological characterization is unique and allows us to more effectively develop advanced surface modification treatments tailored to specific applications. Another strength is our Section's ability to characterize microstructural and surface chemistry evolution of materials and coatings exposed to tribological environments.

Coating Process Development

Projects in this area are aimed at exploring the factors that control the tribological properties of surfaces given surface-modifying treatments such as ion-beam-assisted deposition (IBAD), ion beam deposition (IBD), microwave and plasma-assisted chemical vapor deposition, and plasma-sputter deposition. The major emphasis is on developing low-friction, highly wear-resistant surfaces that can endure extreme tribological conditions due to high loads, speeds, and temperatures. We have developed a variety of low-friction, wear-resistant coatings and deposited them on metals, ceramics, and polymers intended for utilization in components for low-heat-rejection diesels and other high-temperature advanced engines and engine power trains. Specific examples include the development and testing of diamondlike carbon coatings (DLCs) on polymers and ceramic parts under conditions of high loads, speeds, and temperatures; development of smooth, nanocrystalline diamond films on ceramics for machining and sliding wear applications; development of naturally occurring lubricious-oxide layers on superalloys for gas turbine applications at temperatures to 1000°C; development of wear-resistant boride layers on steels and other alloys. More recently, we have made significant advances in the development of DLC films (which we now call NFC coatings) produced by plasma-assisted CVD that exhibit extremely low friction coefficients (<0.001) under dry sliding conditions. Response from industry on the NFC coatings has been tremendous, and currently the majority of our efforts focus on NFC coating development and assessment.

Our coating processing research focuses on two broad deposition techniques: development of low-friction and low-wear surfaces by a variety of surface-modification methods, including physical-vapor-deposition techniques (ion-beam-assisted deposition, plasma sputtering, and thermal evaporation), and development of ultrahard, low-friction diamond, diamondlike, and other similar coatings by plasma-assisted chemical vapor deposition.

Chemical Vapor Deposition Coatings

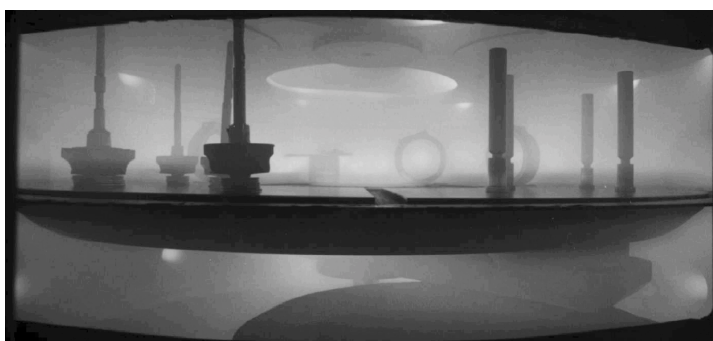
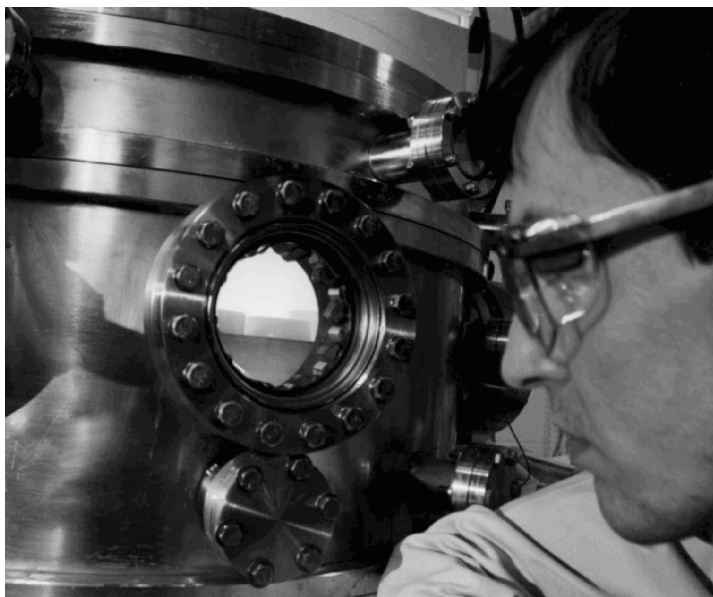
Over the past 36 months, the Section's coating development effort has focused primarily on the use of plasma-assisted chemical vapor deposition (PACVD) to deposit NFC coatings. The Section has two facilities for CVD of these coatings: a PACVD unit and a microwave PACVD unit. The microwave PACVD system was procured for the study and development of polycrystalline diamond (PCD) films. While the microwave plasma deposition source allows production of diamond, NFC, and similar ultrahard coatings on ceramics and other high-temperature materials, the main thrust of the R&D on this system is production of smooth, adherent crystalline diamond films that serve as lubricious, wear-resistant coatings without causing severe wear of mating components. Initial efforts were directed at forming smooth, thick diamond films at high deposition rates of up to 1 $\mu\text{m/hr}$. While a multistep microwave-plasma-deposition process was developed to deposit relatively smooth diamond films on rough-surfaced films, the Tribology Section is now teamed with groups in ANL's Chemistry Division that have developed microwave PACVD processes using Fullerene-rich Ar gases as precursors for the formation of nanocrystalline diamond (NCD) films. The Fullerene-based films have a much finer microstructure than conventional PCD films grown from CH_4/H_2 plasmas and consequently have better tribological properties.

The second PACVD facility is actually a plasma-sputtering deposition system that was designed as a PVD system, but is now being used in a PACVD mode (see next figure) using an RF-etch capability. This system consists of a 24-inch-diameter vacuum vessel set up for diode (RF-diode, RF-magnetron, or DC-magnetron) sputter deposition from three different target locations. This system is being utilized, because in general, PACVD can be scaled up much more readily to handle large quantities of parts than can IBAD or microwave assisted CVD, where the technology is not as mature. Recent R&D efforts on this system have shown that very high-quality NFC films can be achieved and that the tribological properties can be effectively tuned and optimized for particular conditions by controlling the gas chemistry.

Response to the NFC technology has been phenomenal. More than 3,500 inquiries have been received on this technology, and we have established more than 85 nondisclosure agreements with private industry, as well as some 30 small projects with companies to treat small numbers of prototype components for field tests. Some of the field tests have already been successful, and we are now working with several firms to demonstrate that this technology can be scaled up to the commercial-size coating systems required for high-volume production.

Physical Vapor Deposition Coatings

When the Tribology Section was organized 10 years ago, its primary focus was on generic long-term research on advanced surface modification processes to improve the friction and wear performance of materials used in energy-intensive sectors such as transportation. The coating research originally focused on physical vapor deposition (PVD) processes with major emphasis on ion-beam-assisted deposition (IBAD) processes. This facility consists of a large, ultrahigh-vacuum chamber with two individually controlled electron-beam evaporation sources and simultaneous or sequential ion sources that produce ions with energies from a few eV up to 1500 eV. Materials that can be evaporated include Ag, Al, B, Ba, Ca, Cr, Cu, Fe, Mg, Mo, Nb, Ni, Pt, Sn, Ti, and Zn, either singly or in combination. Various types of specimen holders enable



Plasma-sputtering deposition system

rotational motion, ensuring uniformity in the coating. Parts as large as engine pistons can be coated. In addition, the Section has a three-target sputtering apparatus for the deposition of multilayer coatings comprising Si, Au, B, C, Al, Ti, Cr, Fe, Ni, Cu, Mo, Ag, W, Pt, or Al_2O_3 . An ultrahigh-vacuum apparatus for depositing Fullerene compounds as tribological coatings has recently been built, as has an apparatus for exposing test specimens to fuels and solvents.

Previous research activities using the IBAD facility included efforts to deposit adherent silver coatings on oxide, carbide, and nitride ceramics to improve the friction and wear at elevated temperatures. The results were very impressive; reductions in wear rate ranged from factors of 10 to 3500, depending on load, temperature, velocity, and substrate. We also developed and tested soft metallic coatings on ceramics and piston ring/liner assemblies under conditions that simulate actual engine operation. Tests with synthetic liquid lubricants show a synergistic effect on both wear and friction, with improvements in both properties when a liquid lubricant is used in conjunction with a silver-coated ceramic. With the simultaneous use of silver and synthetic oil, the friction coefficients were reduced by factors of 3 to 10 from that with oil alone, depending on test temperature (e.g., from 0.15 to 0.05 at room temperature, and from 0.13 to 0.015 at 300°C). Furthermore, in most cases, wear was reduced to unmeasurable levels even during tests at high temperatures and loads.

Currently, the IBAD system is used for more routine nontribological applications where specialty coatings with excellent adhesion are needed. It is now being used primarily by the Ceramics Section to investigate the role of directed energetic ion beams in promoting the growth of films with preferred orientations and that serve as substrates for high-temperature superconductors. This application appears to be successful, and our Section recently designed and fabricated a second IBAD chamber with low-energy electron diffraction capabilities to study the evolution of the film orientation during growth. In these activities, the IBAD chambers are used to produce buffer, or “template,” layers for superconductor development. Yttria-stabilized-zirconia biaxially textured layers are deposited onto metal substrates, which are then capped with evaporated cerium oxide layers. The tape is then coated with a superconducting material. The effect of the ion beam is studied for producing oriented layers. Studies are also performed on magnesium oxide during inclined-substrate deposition for the same goal. The IBAD technique is also being used to deposit Pt bottom electrodes on Si. Top electrodes are also deposited on wafer stacks of (Ba, Sr)TiO₃ by the MOCVD technique. This collaboration is jointly with ET (Office of Transportation Technologies - Capacitors) and MSD (DARPA - Frequency Agile Materials).

In other areas, SiO₂ was deposited on diamond for a Chemistry Division project on MEMS lithography; ion-sputter-smoothing experiments on diamond crystals were performed for an APS project in the User Program Division. In the area of NMR, Au was deposited on Si for self-assembled monolayer studies in a Chemical Technology Division project; finally, Zr was deposited on V-15Cr-5Ti in an ET project involving insulator-layer coatings for fusion reactors.

Tribological Testing and Surface Characterization

The Tribology Section has developed a number of specialized benchtop systems to evaluate and characterize the friction and wear properties of materials and components under a range of operating conditions. These facilities (summarized in the following table) include seven unidirectional pin-on-disk tribometers and three reciprocating pin-on-flat tribometers. The Section is also procuring/designing four additional tribometers to evaluate NFC coatings. The unidirectional pin-on-disk tribometers can operate at up to 1000°C in environments ranging from lab air to inert (e.g., dry nitrogen and argon) to liquid (e.g., water, oil, diesel fuel, and gasoline) lubrication at rotational speeds up to 4000 rpm. The reciprocating pin-on-flat tribometers operate at frequencies ranging from 1 to 2 Hz up to 500 to 1000 Hz, at temperatures ranging from room temperature to 900°C in environments including air, inert gases, liquids (oils, gasoline, diesel fuel, and pressurized gases).

Additional facilities to characterize the mechanical properties of surfaces include a low-load microhardness tester, coating adhesion measurements, surface profilometers, optical and scanning electron microscopy, and more recently, a high-temperature extensometer to monitor thermal bowing in laminated materials arising from differences in thermal expansion properties.

Together, these rigs and characterization capabilities are used to evaluate the friction and wear properties of materials and components under a wide range of conditions that simulate tribological conditions in advanced systems. The characterization capabilities are used extensively to evaluate and identify the physical mechanisms that contribute to friction and wear of materials and components and thereby develop strategies to improve the tribological performance.

ANL Friction and Wear Test Facilities

System	Motion	Configuration	Temperature	Environment	Program
CSEM	Unidirect ^a	POD ^c	Room Temp. (RT)	air, inert gas, liquids	basic R&D
C-P	Unidirect ^a	POD ^c	to 650°C	air, inert gas, liquids	basic R&D
ISC	Unidirect ^a	POD ^c	to 600°C	air, inert gas, liquids	basic R&D
CSEM	Unidirect ^a	POD ^c	to 800°C	air, inert gas, liquids	basic R&D
ANL-1	Unidirect ^a	thrust washer	RT	air, fuels	gas bearing
Falex	Unidirect ^a	BOTD ^d	RT	air, oil, fuels	fuels lubricity
Allison	Unidirect ^a	3POD ^c	to 1000°C	air	gas turbine regenerator core seals
Tranergy	Unidirect ^a	ring-on-ring	RT	air/lubricant	rail lubricants
MT-1	Reciproc ^b	POF ^e	to 900°C	air, oil	gears
C-P	Reciproc ^b	POF ^e	to 300°C	air, inert gas, oil, fuels	engine and fuel system components
Falex	Unia/Rec ^b	POD/POF	RT	air, inert gas	basic R&D
Shaft Seal	Unidirect ^a	shaft seal	RT	inert, water	shaft seals
Metal Forming	Metal Stamping		RT	air	metal-forming lubricants
ANL-1	Unidirect ^a	thrust and journal bearing	RT	air	gas bearing
ANL-2	Reciproc ^b Fretting	POF ^e	to 100°C	air, inert gas, fuels, pressurized gases	fuel system components
Falex Multi- Specimen	Unidirect ^a	4-Ball	RT	air, oil, fuels, pressurized gases	fuel lubricity
ANL-3	Unidirect ^a	POD ^c	RT	vacuum	aerospace and MEMS

^aUnidirectional.^bReciprocating.^cPin on disk (POD).^dBall on three disc (BOTD).^ePin on flat (POF).^fSystems being procured and/or designed and fabricated.

R&D Activities

The facilities described above are used in support of a number of R&D activities, including efforts on development of NFC coatings for fuel systems, development of non-sulfur-based fuel additives, demonstration of NFC coating processes on commercial coating systems, characterization of NFC coating uniformity, characterization of NFC coatings, assessment of material needs for fuel cell air compressors, characterization of boundary layer lubrication mechanisms, characterization of EGR soot effect on lubricant properties, characterization of laser glazing to reduce friction and wear of rail steels, characterization of rail lubricants, development of boric-acid-based metal-forming lubricants, and development of superplastic joining of ceramics. Highlights of these activities are presented below:

Characterization of NFC Coating Properties

The mechanical and tribological properties of ANL's near-frictionless carbon (NFC) coatings are dependent on the plasma-assisted chemical vapor deposition (PACVD) process conditions — most notably substrate bias and process gas composition. Changing the ratio of the process gases used during deposition has a significant impact on the friction and wear properties.

To better understand why the properties of the coatings vary with process conditions, several characterization techniques are employed at ANL to determine the microstructure, composition, and bonding in the different types of NFC coatings. These techniques include (a) scanning electron microscopy (SEM) to characterize the morphology of as-deposited coatings and wear-tested coatings, (b) transmission electron microscopy to characterize the microstructure of as-deposited NFC coatings and NFC wear-debris (obtained from wear-tested samples), (c) Raman spectroscopy to examine the chemical bonding (in particular, sp^2 bonding) of NFC (and commercial amorphous and diamondlike carbon) coatings, (d) Fourier transform infrared (FTIR) spectroscopy to examine C-H bonding in as-deposited coatings, (e) nuclear magnetic resonance spectroscopy to characterize the hydrogen present in the coatings, (f) hydrogen forward scattering to quantify the amount of hydrogen and carbon present in the films, and (g) nano-hardness measurements to evaluate the hardness and modulus of NFC coatings.

Microstructure

The next figure is a cross sectional TEM photomicrograph of an NFC-6 film deposited on a single-crystal silicon wafer. The Si-oxide layer is a bond coating that is required to form NFC coatings on a wide variety of materials — there are only a few materials on which NFC will form without the use of a bond coating. In comparing the NFC-6 film with other NFC films, we noted that the NFC-6 films had the lowest level of internal stresses and that significant levels of oxygen were present. Selected-area diffraction of the NFC coating revealed that it was indeed amorphous — no indication of crystallinity was observed.



Cross section of NFC-6 film deposited on single-crystal silicon wafer

Bonding

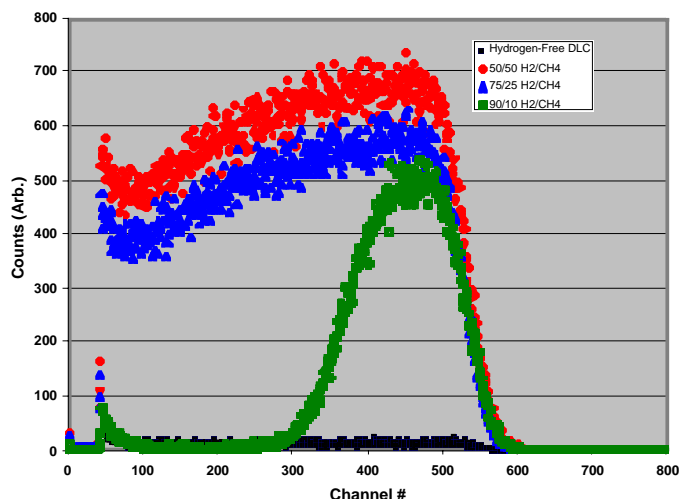
Raman spectroscopy has been used extensively to characterize the C-C bonding of NFC coatings. This information is then coupled to quantum-chemical modeling (conducted by the Materials Science Division) to predict the structure of NFC coatings. The ANL process produces films that are unique in respect to the amount and size of sp^2 ring distribution (especially NFC-6 with an I_d/I_g ratio of ≈ 0.5 and a peak spread of 213 cm^{-1} with a 1460 cm^{-1} center position average). This is believed to be due to a unique combination of many sp^2 disorder rings in the small to mid-size range. This work suggests that the backbone structure of NFC consists of small, dispersed, highly strained sp^2 carbon ring clusters in a matrix of predominantly sp^3 carbon, with the best inert-atmosphere friction performance exhibited by the films with the highest degree of cluster strain. Analysis of FTIR data showing that the majority of the carbon-hydrogen bonds are between hydrogen and sp^3 carbon, together with the hardness/modulus, suggests that the sp^3 matrix (for NFC-6) is soft and polymeric, containing more carbon chains and less cross-linked carbon bonding.

Composition

Typical NFC runs use different levels of H_2 in the process gas to produce NFC coatings with different tribological properties. To differentiate between hydrogen arising from CH_4 molecules from hydrogen originating from H_2 in the process gases, a series of deposition runs with various combinations of D_2 (molecular deuterium) and CD_4 (deuterated methane) was conducted. Hydrogen forward scattering (also known as forward recoil scattering) was subsequently used to quantify the amount of hydrogen trapped in the films.

The next figure shows the forward recoil spectra obtained from three different NFC films produced with 50/50, 75/25, and 90/10 mixtures, respectively, of H_2/CH_4 in the NFC process gas, and a hydrogen-free DLC film. As the hydrogen content in the process gas increases, the concentration of hydrogen trapped in the NFC coatings decreases — a trend opposite to that expected. However, such a trend has been observed in studies on the deposition of amorphous silicon films from hydrogen-silane mixtures in which the decrease in trapped hydrogen with increasing process gas hydrogen was attributed to the formation of volatile SiH_4 species on the surface when neutral plasma hydrogen species reacted with surface SiH_3 radicals.

Analysis of the HFS studies with various hydrogen isotopes in the source gas indicates that the total amount of hydrogen (e.g., H or D) trapped in the NFC films ranged from 32 to 45 at.%, with the highest value of H occurring with a 100% methane process gas. However,



*Forward recoil spectra from three NFC films
and a hydrogen-free DLC film*

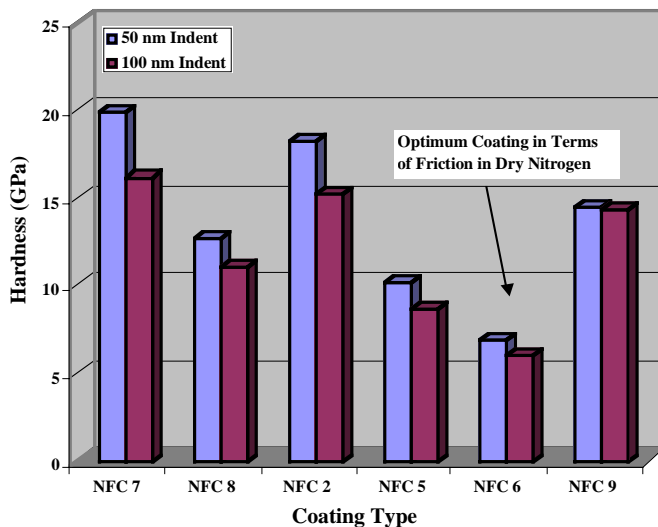
when pure CD_4 is used, the deuterium concentration drops to 32 at.%. With 50/50 or 75/25 gas mixtures, the results appear to indicate that the majority of the hydrogen trapped in the films originates from hydrogen associated with CH_4 , not H_2 . These results are summarized in the table below.

Comparison of Hydrogen Isotopes Trapped in NFC Coatings

Process Gas	H Concentration (at.%)	D Concentration (at.%)
50/50 H_2/CH_4	39	–
50/50 D_2/CH_4	37	4
50/50 H_2/CD_4	16	21
75/25 H_2/CH_4	39	–
75/25 D_2/CH_4	30	9
75/25 H_2/CD_4	23	14

When H_2 is replaced with D_2 , and normal methane (CH_4) is used, the H content in the films remain relatively the same; however, when H_2 is used, but the normal methane is replaced with deuterated methane (CD_4), the level of H drops (although the missing H is replaced with D).

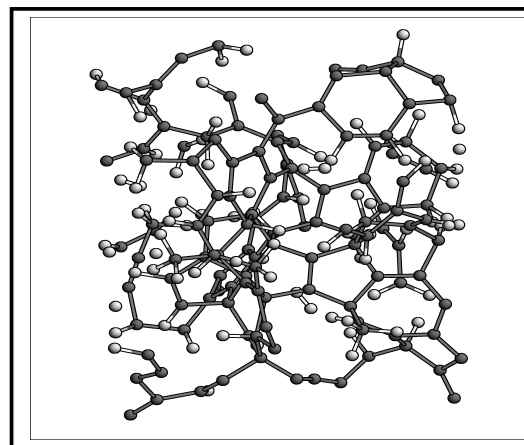
Characterization of the fundamental properties of NFC films will continue. Information on the microhardness of these coatings suggests that the optimum coating (NFC-6) in terms of friction is not the hardest of the NFC family of coatings. In fact, the data (see next figure) indicate that the optimum coating (in terms of friction and wear resistance — in dry N_2) is the softest of this class of coating. These results suggest an alternative approach to minimizing wear. Rather than producing the strongest film to minimize wear, reducing the transfer of friction forces (by lowering the friction coefficient) can be equally effective.



Hardness of various NFC coatings

Structure Modeling

The structure of amorphous hydrogenated carbon having a density and hydrogen content similar to those of the NFC has been studied by using density functional-based tight-binding molecular dynamics. The simulations were done using a periodic cell of about 200 atoms with a hydrogen content of 40%. Density ranged from 1.2 to 2.0 g cm⁻³. The structure of one of these simulations (1.6 g cm⁻³) is shown in the adjacent figure. The sp³:sp²:sp ratio was found to be very dependent on the density of the material. In addition, at some densities, there are cavities that contain H₂ molecules. We are currently working on simulating the surface structure of the amorphous hydrogenated carbon having conditions similar to the carbon found at ANL. Comparison with experimental results will help determine the reason for the very low friction properties of this material.



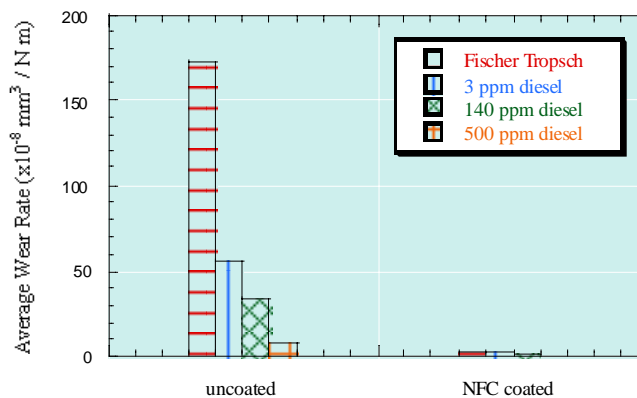
Structure of simulated amorphous carbon having a density of 1.6 g cm⁻³

Evaluation of NFC Coating Compatibility with Diesel Fuels

Current diesel fuels contain large amounts of sulfur (up to 500 ppm) to prevent friction and wear on sliding surfaces of fuel injectors and pumps. However, sulfur causes toxic emissions and degrades the performance of engine after treatment devices. Various government agencies are now calling for the reduction of sulfur in diesel fuels to 40 ppm by 2004 and to 15 ppm by 2007. Such a move will quickly lead to severe scuffing wear and catastrophic failures of the diesel fuel system components. Therefore, there is an urgent need for the development of new materials and coatings that can prevent such failures.

Our approach is to apply ANL's NFC film on fuel injector components intended for use in low-sulfur diesel fuels. When developed and implemented successfully, this approach will have the potential to reduce or eliminate toxic emissions. These activities are supported by DOE's SC-LTR and OTT/OAAT Programs under CRADAs with the Diesel Technology Company, Stirling Thermal Motors, and Front Edge Technologies.

The adjacent figure shows the average wear rates of steel flats that were tested in normal diesel fuel (500 ppm sulfur), low-sulfur diesel fuel (140 ppm sulfur), ultra-clean diesel fuel (3 ppm sulfur), and synthetic diesel fuel (Fischer-Tropsch) with zero sulfur. Results from uncoated test pairs further confirm the pivotal role of sulfur in diesel fuel lubricity. The greatest wear was on flats tested in Fischer-Tropsch fuel (with zero sulfur), while the least wear was in normal diesel fuel containing 500 ppm of sulfur.



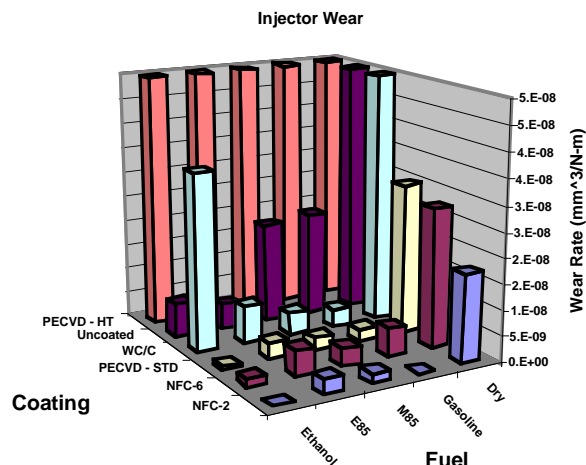
Average wear rates of steel flats tested in diesel fuels with a range of sulfur content

Application of NFC Coatings to Gasoline Fuel Systems

While advanced fuel systems are under development for diesel engines to permit the use of emission control strategies, advanced direct-injected fuel systems for spark-ignited engines are also being developed to improve the fuel economy of gasoline engines. In contrast to current port-injected systems that inject at 4 to 5 bar pressure, direct-injected gasoline fuel systems will inject gasoline at pressures up to several hundred bar, and thus will require high-pressure fuel pumps to supply fuel to the injection system. Furthermore, reformulated gasoline often uses compounds that readily corrode conventional fuel system materials, thus requiring the use of corrosion-resistant materials (e.g., stainless steels) that are prone to scuffing at high loads and stresses.

These activities (supported by DOE's Office of Advanced Automotive Technologies) is evaluating the performance of commercial coatings and ANL's NFC coatings exposed to different grades of gasoline. If successful, this approach will provide a cost-effective solution to implementation of direct-injected gasoline engines.

The next figure shows short-term (e.g., 2000 sec) wear rates of prototypical fuel injector tips (coated and uncoated) subjected to high-frequency reciprocating sliding motion under prototypical loads. The wear rates are strongly dependent on the environment (e.g., dry, gasoline, ethanol, E85, or M85) as well as the type (or presence) of coating. The two ANL coatings (NFC-2 and NFC-6) have slightly different properties, with NFC-2 typically being preferred for air environments, while NFC-6 has optimum behavior in inert environments.



Short-term wear rates of prototypical fuel injector tips using various fuels

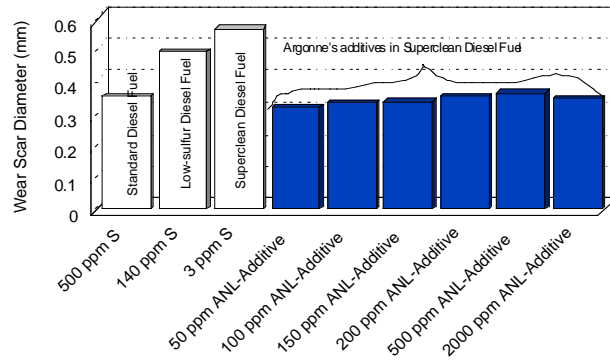
Our future plans for this activity include performing a series of long-duration wear tests in conventional and reformulated fuels with and without lubricity-additive packages. Our focus will also be broadened to examine corrosion behavior and will include high-pressure fuel pump components and seals.

Development of Lubricity Additives for Low-Sulfur Fuels

Current diesel fuels may contain up to 500 ppm sulfur, which causes high levels of SO_2 and particulate emissions. Sulfur also poisons the vehicle pollution control and aftertreatment devices. This has prompted DOE, EPA, and other government agencies to impose tough emission quotas for 2004 and propose even tougher standards for 2007. The impact of all these mandates will fall heaviest on diesel fuel-injection components, particularly injectors and fuel pumps. The major goal of this project is to develop lubricity additives for low-sulfur or even sulfur-free diesel fuels and to demonstrate their viability and effectiveness in controlling friction and wear of fuel delivery systems.

Based on our extensive research on lubricants and additives, internal ANL funds were provided to investigate whether some inorganic compounds can be reformulated and prepared as an additive for low-sulfur diesel fuels. Specifically, we believed that the highly polar nature of these additive molecules should bond strongly to the sliding surfaces of fuel-pump and injector systems and protect these surfaces against friction, wear, and scuffing.

The next figure shows the performance of an Argonne additive (designated as ANL) on fuel lubricity. Standard diesel fuel with 500 ppm sulfur exhibited the highest lubricity among all the fuels tested and thus caused the least wear on sliding test pieces. Low-sulfur diesel (140 ppm sulfur) caused moderate wear. However, the super-clean diesel fuel caused the highest wear and thus had the lowest lubricity. However, as is obvious from the figure, addition of a small amount of ANL's additive (i.e., as low as 50 ppm) had a huge positive impact on lubricity of the super-clean diesel fuel. In fact, the reduction in wear scar diameter is rather remarkable and surpasses the level of reduction that can be achieved by a standard diesel fuel containing 500 ppm sulfur. This means that adding 50 ppm of ANL additive to super-clean diesel fuel will restore its lubricity.

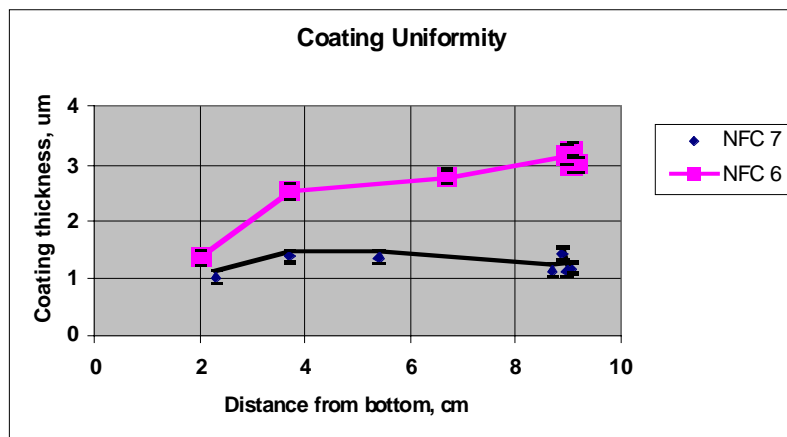


Performance of ANL additive on diesel fuel lubricating

Characterization of NFC Coating Uniformity

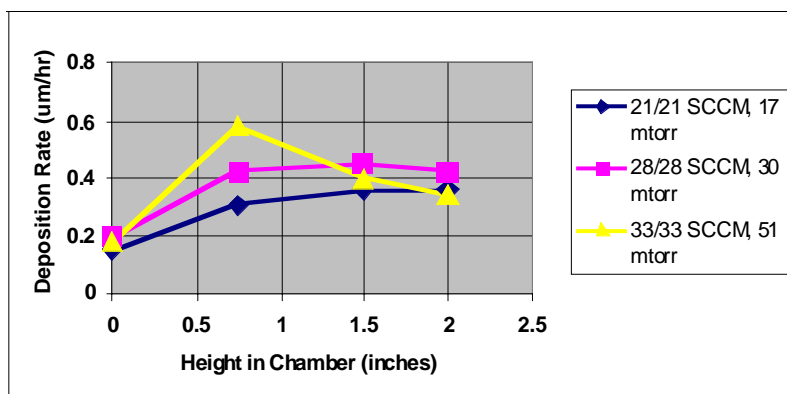
Coating uniformity in terms of coating thickness is a critical concern for most applications. This is particularly true for fuel system applications where high injection pressures (e.g., up to 3 kbar) require clearances in the range of 1 μm between mating components. With this tight tolerance, it is therefore crucial that we can (a) reproduce a given coating thickness from run to run, and (b) produce coatings of uniform thickness on a given component, as well as produce uniform coatings on different components within a given batch.

In the past year, we have developed the proper protocols to deposit coatings on fuel injector plungers with a high degree of top-to-bottom uniformity (next figure) and have provided coated plungers to the Diesel Technology Company for their tests. Initial results looked promising.



Top-to-bottom uniformity of ANL's NFC coatings

As part of this uniformity study, we have also begun pressure/spacing/height tests to establish the effect on coating thickness of deposition pressure, spacing between plungers, and height in the chamber. Initial results show a nonlinear relationship between deposition rate and chamber pressure (see next figure) and a convoluted relationship between deposition rate, pressure, and position in the chamber. These results were determined from flats at the



Deposition rate vs. height in chamber

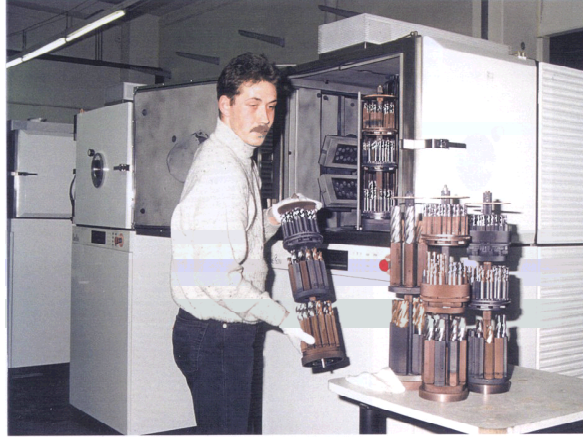
specified heights in the chamber. In addition, there is some indication that below a minimum distance of ≈ 1 to 1.5 cm between plungers, any NFC deposited on a plunger is etched off by hydrogen in the plasma. This has some ramifications for chamber packing in a project now underway to demonstrate that the NFC process can be scaled up for commercial applications.

Scalability of NFC Process

The NFC coating technology developed at ANL has great potential in a wide range of tribological applications. Extremely low friction coefficients coupled with high wear resistance make the coatings attractive for many uses, among them transportation (e.g., fuel systems, engine bearings, valve-train components, gears). Although field trials on NFC-coated components have shown that these coatings are effective, these trials were performed on components coated in a research-grade PECVD coating system. To successfully integrate the NFC coating process into production, it is critical that we demonstrate that the process can be used on large-scale PECVD systems. While PECVD is routinely used to treat components used in heavy-duty transportation applications (such as in plasma-nitriding of fuel injector components), the NFC process conditions are sufficiently different (e.g., process pressure and substrate bias) that we have established a CRADA with CemeCon, Inc., to demonstrate that the process can be adapted to that firm's line of coating systems.

The next figure shows the system that will be used to demonstrate that the NFC process is adaptable to commercial-sized coating systems. Preliminary cost estimates indicate that two of these units could treat more than 2 million heavy-duty fuel injector plungers per year at a cost competitive with current technology.

We are currently procuring a comparable system. Preliminary tests at CemeCon, using NFC coating protocols, indicate that the quality of the coatings (in terms of friction) is comparable to those obtained with ANL's research unit. Our future efforts in this project (supported by the DOE's Office of Advanced Automotive Technologies, and the State of Illinois) will involve collaborative efforts with industrial partners to address coating uniformity issues (in terms of coating thickness and quality) on prototypical components.



Demonstration system to show suitability of NFC process at commercial scale

Low-Friction Materials for Fuel Cell Air Management Systems

Fuel cells require a clean flow of air to the fuel stack, typically supplied by a lightweight and compact air compressor/expander. Effective lubrication of critical components in the compressor/expander is necessary for maximum efficiency and reliability. Conventional grease and oil lubricants are undesirable because they can contaminate the fuel cell stack. Materials and surfaces with low-friction attributes are required for various air compressor/expander components.

The objective of this activity (sponsored by the DOE Office of Advanced Automotive Technologies) is to develop and evaluate low-friction, wear-resistant coatings and materials for key components being developed for the fuel cells. This collaborative project with four companies (Honeywell, Meruit, Vairex, and Mechanology) that are designing experimental air compressor/expander systems for DOE's fuel cell program is structured to identify critical components in the various compressors/expanders being developed for the DOE fuel cell program, apply ANL's NFC coatings to appropriate components, evaluate component and coating performance under prototypical operating conditions, and develop and evaluate self-lubricating polymer composite materials.

Laboratory friction and wear screening test have shown that ANL's NFC coating will provide adequate lubrication for Meruit's air bearings. NFC-coated air bearing components are currently being tested on air bearing hardware, and the preliminary results are excellent.

Test are also in progress to evaluate and develop materials for various components of Mechanology's torroidal intersecting vane machine (TIVM) concept for a compact and lightweight compressor/expander. We have built a new high-speed test rig for this evaluation.

Our future activities in this project will continue the efforts with Meruit to characterize the performance of NFC coatings for air bearing applications, provide NFC-coated components for air foil bearing applications, and perform fundamental friction and wear studies for Mechanology's TIVM concept.

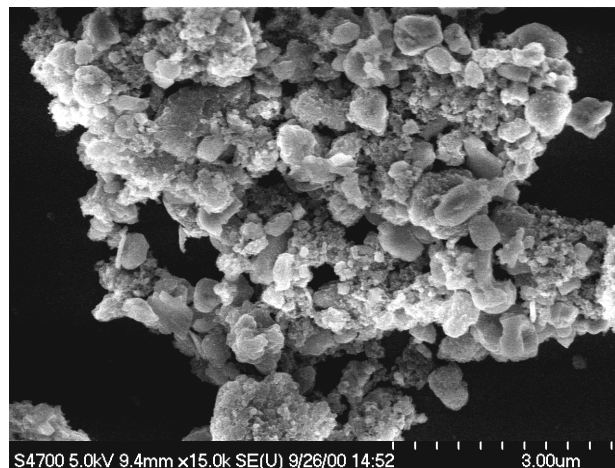
Engineered Surfaces for EGR in Diesel Engines

To meet the impending emission regulatory requirements for direct-injection diesel engines, the use of exhaust gas recirculation (EGR) technology is now believed to be a necessity, although it will have a detrimental effect on engine durability. Increased soot loading and total acid number (TAN) of engine oil operating with EGR will result in significant increases in abrasive and corrosive of major engine components and systems such as piston ring/cylinder liners, valve train components, and main bearings. For successful application of EGR, component surfaces capable of withstanding the more severe condition of abrasive and corrosive wear are required.

The main objective of this project (sponsored by DOE's Office of Advanced Automotive Technologies) is to develop surface technology that will mitigate the deleterious effects on durability of components operating in the aggressive EGR environment. Our research involves efforts to assess the impact of EGR exposure on the composition and properties of the lubricating engine oil and the wear characteristics of pertinent engine components, develop laboratory bench-top friction and wear test methods to duplicate the wear mechanisms in engine components exposed to EGR, and develop materials and surface coatings technologies to prevent the observed wear mechanisms.

Preliminary analysis of oils exposed to two levels of EGR in diesel engine tests showed that soot content, TAN, and viscosity all increased with length of exposure. It was also observed that the average soot particle size (see next figure) decreases with longer exposure time. Initial wear testing with the contaminated oils showed that wear of steel surfaces is determined by both particle volume (soot or particle content) and particle size distribution. Abrasive wear was the predominant wear mode, followed by mild and oxidative wear. Although total wear is higher with larger particles, the transition to a higher wear rate is determined by particle content.

This effort is continuing (with internal ANL support) to focus on analysis of oils exposed to EGR under various conditions. This will facilitate correlation of the soot content and particle size distribution with EGR exposure time. The chemical and structural natures of the particles will also be characterized.



SEM photomicrograph of particles in diesel engine oil with ≈ 100 hours of exposure to EGR

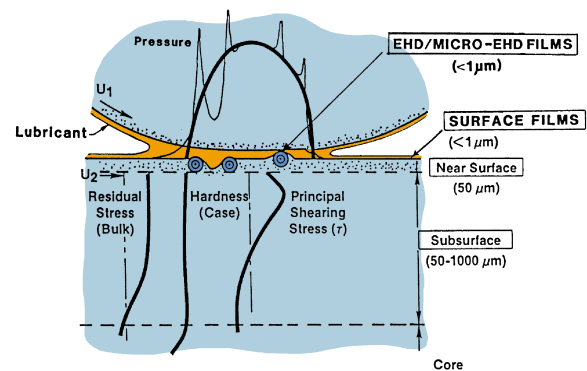
Fundamental Studies of Boundary Layer Lubrication

A vast majority of critical tribological systems and components are oil-lubricated. Due to the inadequate understanding of the basic mechanisms of boundary lubrication and catastrophic failure, an empirical trial and error approach is commonly used to formulate lubricants and develop an effective lubrication strategy. Consequently, prediction of component life and performance is impossible at present. With increasingly stringent and severe operating conditions for lubricated components, the empirical approach is no longer adequate. In order to progress to a knowledge-based lubricant formulation and component performance prediction methodology, a better understanding of the fundamental mechanisms of boundary lubrication and failure is essential.

The ultimate goal of this project is facilitation of effective lubricant formulation and component performance prediction through better understanding of basic boundary lubrication and failure mechanisms. Specific goals are (a) determine the basic mechanisms of catastrophic failure of lubricated surfaces, (b) determine the basic mechanisms of chemical boundary film formation and loss, (c) establish and validate performance and failure prediction methodology for lubricated surfaces, and (d) integrate coating and lubrication technologies to achieve optimum performance enhancement for tribological interfaces.

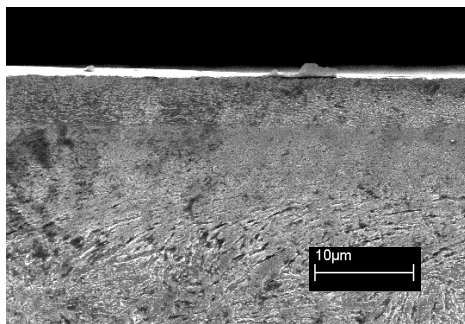
Surface lubrication requires three structural elements (adjacent figure), namely lubricant fluid film, chemical boundary films, and near-surface material. While there is good understanding of the fluid-film element through theory and experiments, the behavior and attributes of the chemical boundary film and the near-surface material are poorly understood. The focus of the current project will be on the two poorly understood elements. Dynamic changes occurring in the near-surface material during failure will be characterized by various techniques, with scuffing used as

a model failure mode (scuffing is the sudden catastrophic failure of lubricated surface accompanied by a sudden rise in friction and temperature; it occurs very quickly, on the order of milliseconds). Kinetics of chemical boundary films formation and loss rate will be studied by in-situ surface characterization in ANL's APS facility. Physical, mechanical, and tribological behavior of the surface chemical films will be measured with a variety of surface probing tools and techniques, such as AFM, nanoindentation, and nanoscratch testing.

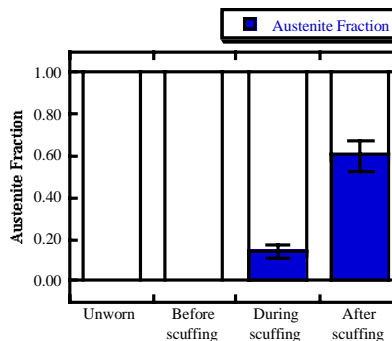


Schematic diagram of the three structural elements of lubrication

Microstructural changes in the near-surface material of hardened 4340 steel blocks used in a ring-on-block test were characterized before, during, and after scuffing failure. Our analysis has also revealed a significant change in the near-surface material. Severe plasticity and grain refinement to a depth of about 20 μm accompanied scuffing failure (next figure [left]). Formation of austenite phase from the original martensite phase was also observed (next figure [right]). A mechanistic model of scuffing based on adiabatic shear instability is currently being formulated.



SEM photomicrograph showing severe plasticity and grain refinement after scuffing



Formation of austenite phase from original martensite during scuffing

Our future efforts on boundary layer lubrication will focus on validation of the adiabatic shear localization mechanism for scuffing, and the characterization (ex-situ and in-situ) of the formation of chemical boundary films from oil additives.

Metal-Forming Lubricant Research

Aluminum and its alloys are highly preferred for use in automotive components because of their light weight, which can substantially increase vehicle fuel economy. It is well known that the surface of aluminum is covered with a thin natural layer of aluminum oxide that renders most conventional lubricants useless and thus leads to adhesion and galling during metal-forming. Surfaces of dies and tools may be abraded by the same harder aluminum oxide layers. To increase the effectiveness of conventional lubricants, flammable and toxic species (such as chlorine, sulfur, and phosphorus) may be added to the fluids. Post-cleaning and/or disposal of these substances is difficult and very costly. Therefore, it is important that new and safe lubricants (that are more suitable for the metal-forming of aluminum and its alloys) be developed.

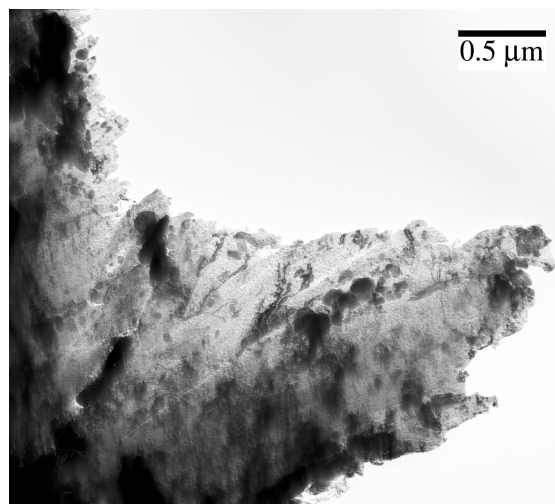
Our approach was to apply ANL's boric acid solid lubricant films on aluminum sheets. Studies conducted in our laboratory demonstrated that boric acid (H_3BO_3), with a friction coefficient of 0.02, is among the best solid lubricants available and is cheap and environmentally friendly. More recent studies revealed that boric acid has a strong tendency to form chemically bonded films on the oxidized surfaces of aluminum and its alloys, and that it can be bonded onto aluminum surfaces from water or methanol solutions of the acid. These findings increase the prospect for developing boric acid as an effective lubricant for aluminum cold-forming.

Laser Glazing of Railroad Track

Laser glazing of railroad steel surfaces has been shown to reduce friction coefficient by up to 40%. This friction reduction is expected to significantly improve rail durability by increasing contact fatigue life. Although the basic mechanism for friction reduction is not fully understood, it is currently attributed to the presence of an amorphous layer on the glazed surface. To optimize laser glazing for railroad applications (the primary goal of this project), the mechanism(s) for friction reduction must be better understood.

This activity entails several efforts to investigate the potential of laser glazing to improve the energy losses associated with flange/track interaction, and to improve the durability of rails and wheels. Benchtop friction and wear tests provide information on the tribological properties of baseline and laser-treated steels, while full-scale wheel/rail rig tests at an Association of American Railroads facility provide information on rolling/sliding wear and energy losses on prototypical components. Preliminary results have demonstrated that proper treatment of rail material (e.g., 1080 steel) can significantly improve friction and wear properties. Because the mechanisms for these improvements are not fully understood, fundamental studies of the microstructure and mechanical properties of as-received and laser-treated rail stock are currently underway.

The adjacent figure is a bright-field transmission electron micrograph of a segment of 1080 rail stock (in plane view) in an as-received state. Analysis of the selected area diffraction pattern of this region indicates that two phases are present: one giving rise to a set of spotty rings and another producing smooth but still sharp rings. The first has been identified as bcc ferrite with an unusually large lattice parameter $a_0 = 2.98 \text{ \AA}$ (ferrite is nominally 2.886 \AA) and coarse grain size (the spotty rings). The second phase is fcc magnetite (Fe_3O_4), with $a_0 = 8.8 \text{ \AA} \pm 0.1 \text{ \AA}$, having finer grain size (the smooth rings); the nominal lattice parameter of magnetite is 8.4 \AA . The unit cell sizes measured here are estimated to be accurate to one part in 60 (or 1.6%), which is not enough to account for the enlarged lattice parameters of these phases relative to their equilibrium values.



TEM photomicrograph of segment of as-received 1080 rail stock

While this research is still in progress, results for the as-received rail stock indicate that the sample consists primarily of ferrite and magnetite and that the grain size of the magnetite is finer than that of the ferrite; they are probably $0.02\text{-}0.03 \text{ }\mu\text{m}$ and $0.1\text{-}0.2 \text{ }\mu\text{m}$, respectively. No significant volume of amorphous phase is present, and the unit cell sizes of the ferrite and magnetite are larger than normal.

Our future work will focus on analysis of laser-treated steel, both in the as-treated and worn states. Tests are also planned to determine if laser-glazing can be used to “heal” cracks in rail stock before gross macroscopic track failure. This research is supported by the DOE Office of Heavy Vehicle Technologies and involves collaborative efforts with industrial laser glazing firms and rail-track maintenance firms.

Top-of-Rail Lubrication Studies

Friction and wear of railroad-car wheels caused by rolling resistance has been the subject of many technical studies in the United States, as has been the larger area of track/train dynamics. Excessive friction between car wheels and rail increases locomotive fuel

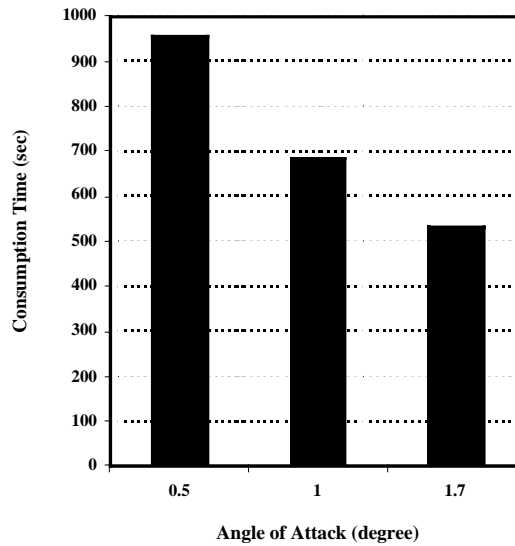
consumption, reduces train speed, and causes damage such as track degradation, wheel and rail wear, and hunting. The hunting phenomenon is caused by disturbing car, track, and traction forces; although the wheels continue at the same rotational velocity, they are on different radii because of coning. Thus, wheels slip on the rail and the axle adjusts to the larger radius; this effect is thereby transferred to the opposite rail and the hunting action continues, causing excessive wear on the rail). To counter the effect of high friction between the rail and wheel, several approaches have been proposed and developed. Current protocols call for the use of lubricants applied to the flanges (of either wheel or track) to minimize frictional forces on the flange surfaces. Recent research has led to the development of top-of-rail (TOR) lubrication schemes in which a degradable lubricant is applied to the top of the rail after the locomotive wheels have passed. This approach is designed to minimize the frictional forces between the wheel and top rail surface that occurs when the wheel “hunts” back and forth, i.e., the wheel rarely is in pure rolling contact with the track, and there is usually some misalignment between the wheel and track so that some degree of sliding occurs in addition to rolling. While the amount of sliding can be small, the energy losses associated with this mechanism can be significant. The TOR lubricant concept is designed to minimize this energy loss.

Field tests on a commercial TOR lubricant concept (developed by Tranergy and now marketed through Timken) that uses a biodegradable lubricant developed by Equilon indicate that the TOR concept can significantly improve the fuel economy of large freight trains. The energy savings, which depend on several field variables, can be quite high (up to 40%). While the TOR lubricant underwent a number of toxicology studies that indicated the lubricant in the as-produced state is environmentally safe, little or no data were available on the environmental nature of the lubricant by-products.

The objective of this research project was to determine if the lubricant by-products were potentially toxic. An experimental wheel/rail test rig was used to simulate stresses and speeds typical of those found in full-scale applications. An enclosure surrounding the interaction zone allowed us to collect and analyze the composition of volatile and liquid TOR by-products. The angle of attack (between the wheel and rail) was adjustable, and a load cell provided continuous monitoring of the top-of-rail lateral forces.

The next figure shows the effect of angle-of-attack (AOA) on the time required to degrade a given quantity of TOR lubricant for an applied load of 5 lb (which simulates a fully loaded rail car), a rotational speed of 300 rpm, and an application of 5 μ L of Equilon's TOR lubricant. As expected, as the AOA increases, the degradation time decreases.

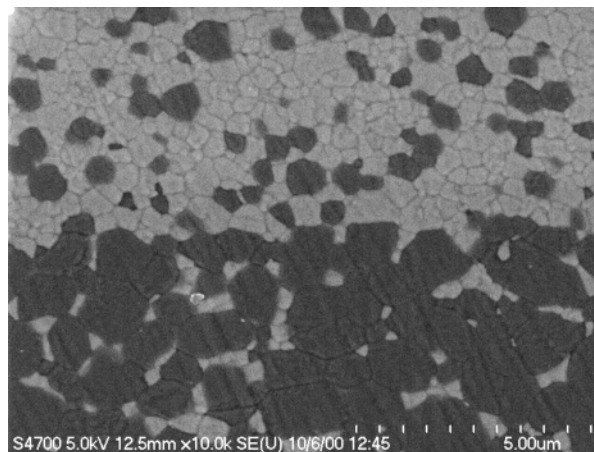
Analysis of the volatile and semivolatile compounds produced as the lubricant was used indicated that in the volatile fraction, the only compounds on the Environmental Protection Agency's (EPA) Superfund List of Analytes detected were contaminants either from the room air or from other potential contamination sources in the laboratory. Similarly, in the semivolatile fraction, none of the detected compounds are on the EPA's Superfund List of Analytes. The major compound in the semivolatile fraction is 1,2-propanediol, which was also found as the major component of the TOR lubricant before testing. Other compounds found in trace quantities either were present in the TOR lubricant or were small fragments from the polymeric component of the TOR lubricant.



Effect of angle of attack on time required to degrade TOR lubricant

Joining of Materials by Superplastic Flow

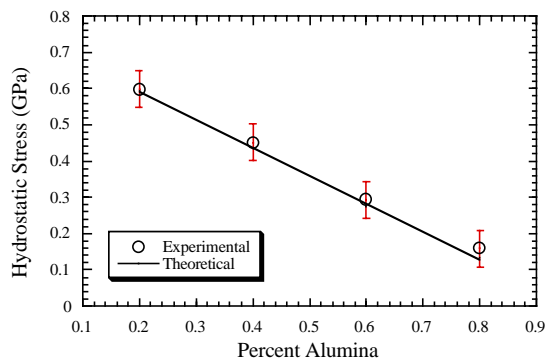
Bonding of ceramics can be quite difficult and is an impediment to the development of many structural components. Diffusional bonding requires high temperatures and highly polished surfaces, while brazing is viable only for low-temperature applications. We have initiated a program on joining materials, initially ceramics, using the principle of superplasticity. If a material deforms superplastically, its grains rotate. Rotation into the material to be bonded can result in strong, pore-free bonds. Our first proof-of-principle experiments were performed on various Al_2O_3 -partially stabilized ZrO_2 composites. A typical pore-free joint between a 20% Al_2O_3 - ZrO_2 and an 80% Al_2O_3 - ZrO_2 is shown below. Vickers indentation tests at the interface have verified that the joint is stronger than either matrix.



Composite containing 20% Al_2O_3 - ZrO_2 (lower) joined to a composite containing 80% Al_2O_3 - ZrO_2 . The joint is pore-free.

We have also made strong joints in the $\text{Al}_2\text{O}_3\text{-ZrO}_2$ system in which residual stresses have been measured by laser fluorescence. A comparison between the calculated residual stresses and the measured stresses is shown in the graph below.

In addition, we have successfully joined an intermetallic (Al-Ti). We are preparing samples for four-point bending tests to determine the bond strength and are now writing a manuscript for Acta Metallurgica. We plan to attempt to bond ceramic-whisker and metal-matrix composites later in 2001.



Hydrostatic internal stresses in alumina phase of composite (circles) as measured from the shift in the peak position of the ruby spectrum compared to the theoretical estimate (straight line) calculated according to the model of Hsueh.

Work-for-Others on NFC

This project is a collection of small WFO contracts with industrial partners to evaluate NFC coatings applied to prototypical components. Approximately 15 small WFO contracts have been established with another 20 to 30 are being negotiated. The main focus of these contracts is the deposition of ANL's NFC coatings on actual parts. After coating, these parts are returned to the industrial partner for evaluation of coating performance.

Program Direction

The Tribology Section plans to continue its close alliance with the transportation sector. There are a number of systems where the technologies and expertise being developed in our Section can be successfully applied. As part of this effort, members of the Section are in close contact with DOE OTT. This effort in the past has proved beneficial in establishing new programs within our Section and we anticipate that it will continue in the future. The Section has also been requested to assist DOE in the development of a multiyear program plan addressing Friction, Wear, and Surface Modification in Transportation. That effort is being supported by the DOE Office of Transportation Technologies. A DOE-sponsored workshop was held at ANL in March 2001 to solicit input from industry on its needs in the area of tribology. The output of this workshop, together with input from site visits to individual companies, will serve as the basis for developing a long-term R&D program between DOE, industry, national labs, and academia.

Recent Work by the Tribology Section

Publications

Tribological Properties of Nanocrystalline Diamond Films

A. Erdemir, G. R. Fenske, A. R. Krauss, D. M. Gruen, T. McCauley, and R. T. Csencsits
Surface and Coatings Technology 120-121 (1999) 565-572.

Friction and Wear Performance of Diamond-like Carbon Films Grown in Various Source Gas Plasmas

A. Erdemir, I. B. Nilufer, O. L. Eryilmaz, M. Beschliesser, and G. R. Fenske
Surface and Coatings Technology 120-121 (1999) 589-593.

A Crystal-Chemical Approach to Lubrication by Solid Oxides

A. Erdemir
Tribology Letters 8 (2), 97-102 (2000).

Self-Replenishing Solid Lubricant Films on Boron Carbide

A. Erdemir, O. L. Eryilmaz, and G. R. Fenske
Surface Engineering, Vol. 15, pp. 291-295 (1999).

Compressive Creep of Polycrystalline ZrSiO_4

K. C. Goretta, T. A. Cruse, R. E. Koritala, J. L. Routbort; J. J. Melendez-Martinez,
and A. R. de Arellano-Lopez (U. of Sevilla)
Submitted to J. European Ceramic Society, in press (2001).

The Boron Oxide-Boric Acid System: Nanoscale Mechanical and Wear Properties

Xiangdong Ma, W. N. Unertl (U. of Maine); and A. Erdemir
J. Mater. Res. Vol. 14, No. 8, pp. 3455-3466 (Aug. 1999).

Nano-Tribological and Wear Behavior of Boric Acid Solid Lubricant

S. Mirmiran, V. V. Tsukruk (Western Michigan U.); and A. Erdemir
Tribology Transactions, Vol. 42 (1999), 1, 180-185.

Low-Friction Coatings for Air Bearings in Fuel Cell Air Compressors

O. O. Ajayi, G. R. Fenske, A. Erdemir, J. Woodford, J. Sitts, K. Elshot, and K. Griffey
Proc. 2000 SAE Intl. Future Car Congress, Arlington, VA, April 12-16, 2000.

Tribological Performance of NFC Coatings Under Oil Lubrication

O. O. Ajayi, M. Alzoubi, A. Erdemir, G. R. Fenske, O. L. Eryilmaz, and S. Zimmerman
Paper presented at 2000 SAE Intl. Future Car Congress, Arlington, VA, April 12-16,
2000; SAE Technical Paper 2000-01-1547.

High-Rate Reel-to-Reel Continuous Coating of Biaxially Textured Magnesium Oxide Thin Films for Coated Conductors

M. P. Chudzik, R. A. Erck, U. Balachandran, Z. P. Luo, D. J. Miller; and C. R. Kannewurf
(Northwestern U.)
Proc. 6th Intl. Conf. on Materials and Mechanisms of Superconductivity and High-Temperature Superconductors, Houston, Feb. 20-25, 2000.

Processing Dependence of Biaxial Texture in Yttria-Stabilized Zirconia by Ion-Beam-Assisted Deposition

M. P. Chudzik, R. A. Erck, M. T. Lanagan; and C. R. Kannewurf (Northwestern U.)
IEEE Trans. on Applied Superconductivity, Vol. 9, No. 2, June 1999, pp. 1490-1493.

Superplasticity and Joining of Zirconia-Based Ceramics

A. Dominguez-Rodriguez, F. Gutierrez-Mora, M. Jimenez-Melendo (U. de Sevilla); R. Chaim (Israel Inst. of Tech.); and J. L. Routbort
Mat. Res. Soc. Symp. Proc. Vol. 601 (2000), pp. 99-104.

Synthesis of Superlow Friction Carbon Films from Highly Hydrogenated Methane Plasmas

A. Erdemir, O. L. Eryilmaz, I. B. Nilufer, and G. R. Fenske
Paper presented at Intl. Conf. on Metallurgical Coatings and Thin Films, San Diego, April 9-14, 2000.

Effect of Source Gas Chemistry on Tribological Performance of Diamond-Like Carbon Films

A. Erdemir, O. L. Eryilmaz, I. B. Nilufer, and G. R. Fenske
Paper presented at 19th European Conf. on Diamond, Diamond-like Materials, Nitrides and Silicon Nitrides, Prague, Sept. 12-17, 1999.

Synthesis of Diamondlike Carbon Films with Superlow Friction and Wear Properties

A. Erdemir, O. L. Eryilmaz and G. R. Fenske
Paper presented at 46th Intl. Symp. of the American Vacuum Society, Seattle, Oct. 25-29, 1999.

High-Dielectric-Constant Ferroelectric Thin Film and Bulk Ceramic Capacitors for Power Electronics

D. Y. Kaufman, M. T. Lanagan (Center for Dielectric Studies, Penn State U.), J. Im, P. Baumann, R. A. Erck, J. Giumarra, S. K. Streiffer, O. Auciello, M. J. Pan (TRS Ceramics), P. Baldo, and J. Zebrowski (New Brunswick Lab.)
Proc. Power Systems World (PSW)/Power Conversion and Intelligent Motion (PCIM) '99 Conf., Chicago, Nov. 6-12, 1999, pp. 317-321.

Creep in Electronic Ceramics (INVITED)

J. L. Routbort, K. C. Goretti and A. R. de Arellano-Lopez (Universidad de Sevilla)
Proc. Intl. Conf. on Mass and Charge Transport in Inorganic Materials, Venice, Italy, May 28-June 2, 2000 (in press).

Effect of Ion-Beam Parameters on In-Plane Texture of Yttria-Stabilized Zirconia Thin Films

T. G. Truchan, M. P. Chudzik, B. L. Fisher, R. A. Erck, K. C. Goretti, and U. Balachandran
Proc. Applied Superconductivity Conference 2000, Virginia Beach, VA, Sept. 17-22, 2000.

Dry Lubricant Films for Aluminum Forming

J. Wei, A. Erdemir, and G. R. Fenske
Paper presented at 1999 STLE/ASME Tribology Conf., Kissimmee, FL, Oct. 11-13, 1999.

Advanced Ceramic Capacitor Technologies: Ferroelectric Thin Films and Metal Electrodes

D. Y. Kaufman, S. K. Streiffer, J. Im, P. Baumann, R. A. Erck, and O. Auciello

Presentation at Passive Components for Power Electronics Workshop, University Park, PA, April 26-27, 2000.

Solid Lubricants and Self-Lubricating Films

A. Erdemir

Chapter 22 for Modern Tribology Handbook, Vol. 2, Materials, Coatings, and Industrial Applications, pp. 787-825, published by CRC Press, ed. B. Bhushan.

Final Report - Top of Rail Lubricant

M. F. Alzoubi, G. R. Fenske, R. A. Erck, and A. S. Boparai

Prepared for the U.S. Department of Energy, Office of Transportation Technologies, and the Office of Heavy Vehicles Technologies, Feb. 2000.

Appendix A: Regular ET Employees

Administration

<i>Staff</i>	R. B. Poeppel, Division Director
	W. J. Shack, Associate Division Director
	R. A. Valentin, Associate Division Director
	A. C. Smith, Building Manager, 212
	A. C. Smith, Environmental Compliance Officer
	P. E. Domagala, Computer Systems Administrator
	M. P. Leyson, Associate Systems Administrator
	J. Nelson, Associate Systems Administrator
	B. H. Malak, Financial Administration
	C. A. Malefyt, Communication Coordinator
	R. R. Fabian, QA Representative
<i>Salaried</i>	B. K. Baudino
	R. N. Lanham
	J. L. Toenies
<i>Hourly</i>	V. A. Martinez
	S. D. Piazza
	E. A. Moravek ^{TRP}

Engineering Technology

	Thermal and Electromechanics	Computational Physics and Hydrodynamics
<i>Staff</i>	J. R. Hull Y. S. Cha S. U. Choi K. E. Kasza T. M. Mulcahy N. T. Obot ⁺⁺	A. M. Hassanein F. C. Chang T. H. Chien H. M. Domanus J. Rest R. C. Schmitt
<i>Technicians</i>	R. K. Smith A. S. Wantroba	
<i>Secretary</i>	J. A. Stephens	J. L. Carlson*
	Sensors, Instrumentation, and Nondestructive Evaluation	Transportation of Hazardous Materials
<i>Staff</i>	A. C. Raptis S. Bakhtiari H.-T. Chien W. A. Ellingson N. Gopalsami E. R. Koehl D. S. Kupperman S. H. Sheen J. G. Sun	Y. Y. Liu R. R. Fabian J. J. Oras V. N. Shah S.-W. Tam
<i>Salaried</i>	R. N. Lanham	
<i>Technicians</i>	C. W. Vulyak, Jr.	
<i>Secretary</i>	J. L. Carlson*	P. Malhotra

Materials Technology

	Ceramics	Corrosion and Mechanics of Materials
<i>Staff</i>	U. Balachandran S. E. Dorris B. L. Fisher K. C. Goretta Y. A. Jee** D. Y. Kaufman R. E. Koritala++ T. H. Lee M. Li** B. Ma J. J. Picciolo J. P. Singh A. Wagh S. Wang** T. Wiencek	K. Natesan O. K. Chopra H. M. Chung S. Majumdar J. H. Park J. Y. Park W. K. Soppet Z. Zeng**
<i>Salaried</i>		J. Franklin
<i>Technicians</i>	J. E. Emerson J. W. Lucas R. L. McDaniel	L. Cairo R. W. Clark T. M. Galvin L. Knoblich D. L. Rink J. C. Tezak
<i>Secretary</i>	S. M. Hagamann	D. K. Moores*
	Tribology	Irradiation Performance
<i>Staff</i>	G. R. Fenske O. Ajayi R. A. Erck A. Erdemir J. G. Hershberger** J. Routbort J. B. Woodford	T. S. Bray M. C. Billone R. S. Daum R. V. Strain H. C. Tsai Y. Yan
<i>Salaried</i>		W. Kettman

Appendix B: Honors, Awards, and Professional Activities

Honors and Awards

M. C. Billone, O. K. Chopra, U. S. Choi, D. R. Diercks, R. R. Fabian, D. Henley, A. B. Hull, K. E. Kasza, Y. Y. Liu, D. Ma, P. Malhotra, D. T. Raske, R. Seidensticker, W. J. Shack, and B. Shelton, Argonne Pacesetter Award for Plant Aging and License Renewal Work at ANL, March 29, 2000.

W. A. Ellingson, E. R. Koehl, G. Forster, and B. Shelton, Argonne Pacesetter Award for extraordinary effort in relocating a facility, March 1, 1999.

A. Erdemir and G. R. Fenske, Argonne Pacesetter Award for Excellence in Achievement in Research that Led to the Discovery of Near Frictionless Carbon Films, Dec. 1998.

U. Balachandran, Fellow of the American Ceramic Society, April 1999.

A. Erdemir, Innovative Research Award for Recent Contributions to Tribology, to be presented at the STLE/ASME Tribology Conf., Orlando, Oct. 12, 1999.

A. Erdemir and G. R. Fenske, Argonne Director's Award, Sept. 28, 1999.

S. Majumdar, J. Franklin, and D. R. Diercks, Argonne Pacesetter Award, Oct. 12, 1999.

L. R. Turner, Citation from G. R. Bachula, Acting Under Secretary for Technology of the U.S. Dept. of Commerce for service to the Partnership for a New Generation of Vehicles, September 1999.

U. Balachandran, Certificate of Recognition and Appreciation for Outstanding Service as an Organizer for the Symposium on High Temperature Superconductors, 2000 TMS Ann. Mtg., Nashville, March 12-16, 2000.

A. S. Wagh and D. Singh, 2000 Federal Laboratory Consortium Award for Excellence in Technology Transfer for the Development of Ceramicrete, Charleston, SC, May 10, 2000.

U. Balachandran, Certificate of recognition as Organizer for Symposium on Materials for Field Agile Microwave Electronics and Next Generation Wireless Telecommunication, 102nd Ann. Mtg. of the American Ceramic Society, St. Louis, MO, April 30-May 3, 2000.

W. A. Ellingson, Certificate of Appreciation for support and commitment as a Mentor for the Institute of Biotechnology, Environmental Science, and Computing, July 1999.

W. A. Ellingson, Certificate of Appreciation for support and commitment as a Mentor for the Energy Research Undergraduate Laboratory Fellowship Program, 2000.

A. Erdemir, Member of 2000 Class of Georgia Tech's Academy of Distinguished Engineering Alumni, induction Oct. 7, 2000.

Y. Yan, Selected participant in "2000 Outstanding Scientists of 20th Century-Second Edition," International Biographical Center, Cambridge, UK.

T. S. Bray, R. Daum, W. Kettman, D. J. McGann, D. P. McGann, R. V. Strain, and Y. Yan, Argonne Pacesetter Award for meeting difficult deadlines and/or demands of a technical, administrative, or sponsor-related nature.

W. A. Ellingson, "Gas Turbine Industry Champion" award presented by the Gas Turbine Assoc. Board of Directors, Annual DOE Gas Turbine Review Meeting, Washington, DC, Dec. 4-7, 2000.

U. Balachandran, Certificate of Appreciation as Symposium Organizer, 2000 Fall Meeting of the Materials Research Society, Dec. 2000.

A. Erdemir, Certificate of Appreciation in conjunction with planning and carrying out Annual Meeting Program, Nashville, May 7-11, 2000, presented by Society of Tribologists and Lubrication Engineers.

Professional Society Activities

A. Erdemir, Member, American Society for Metals.

A. Erdemir, Member and Secretary, Ann. Mtg. Program Committee of the Society of Tribologists and Lubrication Engineers.

A. Erdemir, Member, Sigma Xi Honor Society.

R. C. Niemann, Member, Cryogenic Society of America, Chairman of Midwest Chapter.

R. C. Niemann, Fellow of American Society of Mechanical Engineers.

W. A. Ellingson, Chairman, Inspection Requirements, Task Group on Graphite and Ceramic Pressure Equipment (ASME Boiler & Pressure Vessel Code Committee), May 1996-July 1999.

W. A. Ellingson, Invited Membership, ASME B89 Working Group 4.23 on X-ray Computed Tomographic Imaging Metrology, Feb. 1997.

A. Erdemir, Secretary 54th Annual Meeting of Society of Tribologists and Lubrication Engineers, Las Vegas, May 23-27, 1999.

U. Balachandran, Organizer, 1999 Annual Meeting of TMS, Symposium on High-Temperature Superconductors: Synthesis, Fabrication, and Application, San Diego, Feb. 28-March 4, 1999.

D. Singh, Member, ASME Mixed Waste Committee on Waste Minimization and Processing, August 1994-1998.

J. P. Singh, Symposium Organizer, Innovative Processing and Synthesis of Ceramics, 101st Annual Meeting of American Ceramic Society, Indianapolis, April 25-28, 1999.

J. P. Singh, Symposium Organizer, Thermal Barrier Coatings, 101st Annual Meeting of American Ceramic Society, Indianapolis, April 25-28, 1999.

J. P. Singh, Symposium Organizer, Ceramic Matrix Composites, 101st Annual Meeting of American Ceramic Society, Indianapolis, April 25-28, 1999.

A. Erdemir, Vice Chair, Annual Program Meeting, Society of Tribologists and Lubrication Engineers, 1999.

K. C. Goretta, Chair and Session Organizer, Fibrous Monoliths and Solid Freeform Manufacturing I and II Sessions, 24th Annual Engineering Ceramics Division Meeting, American Ceramic Society, Cocoa Beach, FL, Jan. 23-28, 2000.

A. Erdemir, Chair, Annual Program Meeting, Society of Tribologists and Lubrication Engineers, 2000.

W. A. Ellingson, Session Co-Chair, Solid Freeform Manufacturing - II, 24th Annual Conference on Composites and Advanced Materials, sponsored by American Ceramic Society, Cocoa Beach, FL, Jan. 23-28, 2000;

U. Balachandran, Organizer, Symposium II: High-Temperature Superconductors - Crystal Chemistry, Processing, and Properties, Fall MRS Meeting, Boston, Nov. 27-Dec. 1, 2000.

U. Balachandran, Organizer, Symposium on High-Temperature Superconductors, 2000 TMS Annual Meeting, Nashville, March 12-16, 2000.

U. Balachandran, Organizer, Symposium on Materials for Field Agile Microwave Electronics and Next Generation Wireless Telecommunication, 102nd Annual Meeting of American Ceramic Society, St. Louis, April 30-May 3, 2000.

R. A. Erck, Session Chair, Ceramics and Composites, STLE 55th Annual Meeting, Nashville, TN, May 7-11, 2000.

J. R. Hull, Member, American Physical Society, 2000.

J. P. Singh, Conference Organizer for "Symposium on Innovative Processing and Synthesis of Ceramics, Glasses, and Composites," 102nd Annual Meeting and Exposition of American Ceramic Society, St. Louis, April 29-May 3, 2000.

J. P. Singh, Conference Organizer for "Symposium on Thermal Barrier Coatings," 102nd Annual Meeting and Exposition of American Ceramic Society, St. Louis, April 29-May 3, 2000.

J. P. Singh, Conference Organizer for "Symposium on Ceramic Matrix Composites," 102nd Annual Meeting and Exposition of American Ceramic Society, St. Louis, April 29-May 3, 2000.

A. Erdemir, Chairman, 2001 Annual Meeting Program of Society of Tribologists and Lubrication Engineers, Orlando, May 20-24, 2001.

Other Committee Participation

T. S. Bray, Member, ANL-E Criticality Safety Self Assessment, December 1999 through February 2000.

R. A. Valentin, Member, DOE Steering Committee for University Awards in Nuclear Engineering, 1987 to 1999.

R. A. Valentin, Chairman, University of Chicago Board of Governors Outstanding Service Award Committee, 1984-1999.

U. Balachandran, Ph.D. thesis defense committee, Northwestern U., Evanston, IL, June 9, 2000.

U. Balachandran, Ph.D. thesis supervisor, Northwestern U., 1996-2000.

W. A. Ellingson, Member, M.S. Thesis Committee (Todd Spohnholtz), U. of Illinois at Chicago, May 1997 to June 1999.

R. C. Niemann, Member, Argonne Materials Coordinating Committee (HTS Materials), ongoing.

R. A. Erck, Chairman, Ceramics and Composites Section, Soc. of Tribologists and Lubrication Engineers, May 1999 to May 2000.

A. B. Hull, Member, Moscow Sister City Committee of the Chicago Sister Cities Program, 1997-1999.

P. E. Domagala, Member, Year 2000 Working Group, 1998-1999.

W. A. Ellingson, Member, Ph.D. Thesis Committee (Senol Pekin), U. of Illinois at Urbana, August 1997 to August 1999.

U. Balachandran, Member, Exam. Comm., Ph.D. Qualifying Exam - Northwestern U., April 29, 1999.

K. C. Goretta, Member, M.S. Thesis Defense Committee for T. G. Truchan, IIT, April 6, 2000.

U. Balachandran, Development of Textured Buffer Layer on Metal Tapes for Oxide Superconductors, Reviewer, SBIR Grant Application No. 55208-99-I, June 1, 1999.

U. Balachandran, Meter Length YBCO Coated Conductor Development, Reviewer, SBIR Grant Application No. 55911-99-I, June 1, 1999.

T. S. Bray, Member, PAAA Committee, July 1999-June 2000.

T. S. Bray, Member, Waste Minimization and Pollution Prevention Committee, April 1999-July 2000.

K. C. Goretta, Member, Ph.D. Thesis Defense Committee for B. C. Prorok, U. of Illinois, June 2, 1999.

J. P. Singh, Ph.D. Thesis Advisor for Jae Hong Cheon, IIT, Chicago, 1999.

J. Y. Park, Member, Ph.D. Committee, U. of IL at Chicago, 1999.

Y. Y. Liu, Organizer, 1999 DOE SARP Reviewers Mtg., Argonne, Aug. 17-19, 1999.

U. Balachandran, Ph.D. Thesis Examiner for X. Wang, U. of Wollongong, Australia (Nov. 1999).

K. C. Goretta, Member, M.S. Thesis Defense Committee for B. J. Polzin, IIT, Chicago, Nov. 8, 1999.

W. A. Ellingson, Reviewer, High Technology Proposals for the State of Connecticut, Feb. 2000.

J. R. Hull, Member, NASA Peer Review Team for Space Station Flywheel Project, Jan. 2000.

J. R. Hull, Organizer, 18th Symp. in Energy Engr. Sciences, Argonne National Laboratory, Argonne, IL, May 15-16, 2000.

K. C. Goretta, Member, Ph.D. Thesis Defense Committee for Barton C. Prorok, U. of IL, Chicago, March 10, 2000.

N. Gopalsami, Reviewer, SBIR proposals for DOE National Energy Technology Laboratory, 2000.

N. Gopalsami, Member, Working Group in Science and Technology for Environmental Stewardship's Sensor Initiative Workshop, INEEL, June 19-20, 2000.

A. Erdemir, Member, Ph.D. Thesis Defense Committee for Julien Fontaine, l'Ecole Centrale de Lyon, June 2000.

